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ADVISORY GROUP FOR AEROSPACE RESEARCH & DEVELOPMENT

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on

Thermophysical Properties of Solid Materials
Project Section 1A
Cooperative Thermal Expansion Measurements up to 1000°C

by

E. Fitzer

NORTH ATLANTIC TREATY ORGANIZATION

AGARD Advisory Report No.31

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ADVISORY GROUP FOR AEROSPACE RESEARCH AND DEVELOPMENT

(ORGANISATION DU TRAITE DE L'ATLANTIQUE NORD)

THERMOPHYSICAL PROPERTIES OF SOLID MATERIALS, PROJECT SECTION 1A, COOPERATIVE THERMAL EXPANSION MEASUREMENTS UP TO 1000°C

by

E.Fitzer

(Coordinator)

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FOREWORD

The Structures and Materials Panel of the Advisory Group for Aerospace Research and Development (AGARD) comprises scientists, engineers and technical administrators from government, universities and industry, who are concerned with the advancement of aerospace research and development and with the provision of data necessary for the design and fabrication of the vehicles and equipment which NATO requires. The Panel provides a mechanism for discussion, the exchange of information and for conducting co-operative theoretical and experimental studies in selected areas.

The development of aerospace vehicles and equipment requires solid materials which can withstand increasing temperatures and loads. Typically, gas turbine rotors have to be made in larger sizes and to operate at higher peripheral speeds. Components such as these, and many others, are subjected to thermal distributions under conditions of constraint with consequent deformation and the generation of thermal stresses. Thermophysical properties, primarily heat capacity, specific heat, thermal conductivity, diffusivity and thermal expansion and mechanical properties such as modulus of elasticity, Poisson's ratio and yield point, are involved in these phenomena. All these properties vary with temperature in a non-linear manner.

Thermal stresses, which usually reach a maximum during transient heating or cooling, are superimposed on stresses resulting from external loads, inertia forces and on residual stresses. Varying operational conditions involve cyclic variations of temperature and thermal stress with the possible consequence of low cycle fatigue. This can be as critical a factor as creep behaviour under steady elevated temperature.

The mathematical treatment of thermal stresses is complex and, because of the many non-linear relationships involved, iterative methods of solution have to be employed. Further, because reliable data on the variation of the several properties with temperature are not available, average data have to be used with consequent loss of precision in the theoretical predictions. Under these circumstances, reliable results can only be obtained by resort to expensive and time consuming tests of structural components and assemblies.

The obvious need for reliable data stimulated the Structures and Materials Panel to form a Working Group on Thermophysical Properties in early 1966. At first attention was given to the state of knowledge in the appropriate fundamental and applied sciences as well as to the rapidly developing experimental techniques for temperatures up to 3000°C. In March 1967 Prof. Dr Erich Fitzer reported on the state of the art. As a result, the Panel decided to institute a co-operative programme among interested scientists, in several NATO countries, for the purpose of checking their experimental equipment and procedures by using well defined samples of very pure metals, ceramics, graphites and some engineering materials.

This report deals with the results of the co-operative programme on thermal expansion behaviour of the materials mentioned above in the range between ambient temperature and 1000°C. Beyond 1000°C the experimental technique becomes more difficult; nevertheless the co-operative programme continues and further results are expected in the near future. The work already done has proved very satisfactory and has shown the way by which reliable data on engineering materials for the use of designers can be obtained. These co-operative efforts have also shown that some of the materials investigated are suitable standards for more general use.

The Panel is indebted to Germany and the United States who contributed the excellent sample materials of high quality, some of which were specially produced for these investigations, and to the scientists and specialists in many laboratories who offered their services and time most willingly. Furthermore, the Panel was fortunate in finding an accepted authority in this particular field of science, Prof. Dr Erich Fitzer, director of the Institut für Chemische Technik der Universität Karlsruhe, to act as co-ordinator. The Panel is obliged to Prof. Dr Fitzer, the author of this report, who devoted himself so enthusiastically and conscientiously to the task of encouraging and communicating with the participants of this project. We are also grateful to our Working Group, under the Chairmanship of Prof. Dr F.M.Bollenrath who first suggested this project, to whose capable hands the management of this part of the Panel's work has been delegated.

Anthony J.Barrett,
Chairman, AGARD Structures & Materials Panel

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THERMOPHYSICAL PROPERTIES OF SOLID MATERIALS

E.Fitzer

1. INTRODUCTION

As a part of a major project concerning thermophysical properties of solid materials at high temperatures this is a report on the special section of this project dealing with thermal expansion up to temperatures of 1000°C.

1.1 The Aim of the Cooperative Measuring Programme TX 44 "Thermophysical Properties"

The complete project TX 44 is aimed at determining absolute accuracy of thermophysical property data of engineering materials at high temperatures by means of a cooperative measurement programme. The results are to indicate the best way of obtaining accurate data. This involves clarifying

(1) whether it is best to experimentally determine the properties of a material at the temperatures for which the data are needed,

(2) whether known low temperature values on the material considered should be extrapolated to high temperatures,

(3) whether it is better to derive estimated high temperature data of the material considered by a comparison with a similar material whose data are available from data compilations.

To deal with these questions, it is first necessary to define the terms accuracy, reproducibility, repeatability and precision of measurements as well as the suitability of the materials used in the present comparative measurements.

The absolute accuracy of thermophysical data depends on the suitability of the material as well as on the absolute accuracy of the measurement procedure of both the individual participant and of the cooperating group.

The suitability of the material for cooperative thermophysical measurements is determined by the degree to which the following requirements are satisfied:

(1) The samples must be representative for the considered material.

(2) The sample materials used by the different laboratories and for different measurements must be equal and homogeneous.

(3) The sample materials must not change as a consequence of prolonged or repeated exposure to elevated temperatures as incurred during the measurements.

As to the *accuracy of the measurements* of the individual participant, a distinction has to be made between random errors and systematic errors.

All *random errors* influencing the determination of the length change and the temperature of the sample in both measurement and evaluation can be eliminated by repetitive measurements by the same participant under equal conditions. Customarily the arithmetic mean of repetitive measurements is considered to be the most probable individual result. The band width of scatter of one participant, most exactly characterized by the standard deviation, depends on the measurement precision of all parts of the apparatus used as well as on deviations due to the measurement system, the measurement procedure, and the evaluation.

If it were sure that during repetitive measurements no changes in the system had occurred, then the band width of scatter of the results would characterize the *repeatability*. Inasmuch as such system changes during repetitive measurements carried out by the individual participants cannot be ruled out, although the same apparatus and the same method are used, the band width of scatter is always considered as characterizing the *reproducibility*. In the following report the *individual reproducibility* describes the band width of scatter of results obtained by repetitive measurements of one participant, a small band width indicating high reproducibility.

If a sufficient number of measurements were carried out on the same sample under equal conditions, the average value of the results should be free of any random errors. Repetition of sufficient single runs at some other time with the same apparatus should yield the same average value. This consideration applies to any individual apparatus. It

is also valid for any individual laboratory participating in the programme, provided each laboratory employs but one apparatus. In one case, the same participant has used two different apparatuses. These data are considered as two independent individual results.

To obtain true absolute values it is necessary to know the individual systematic error of the apparatus used. Determination of the systematic error can be accomplished by calibration of the apparatus by means of a suitable standard of known thermophysical behaviour. It must be assumed that such calibrations are routinely carried out by the participants although the procedures employed are by no means uniform.

It was the aim of the comparative measuring programme here reported to investigate to which extent such routine calibration leads to comparable data and which deviations must be reckoned with. Furthermore, an analysis of the results was to show if averaging of results obtained by different laboratories leads to a more accurate value.

A programme of this type can be carried out in two ways:

(1) Round robin tests on the same sample. In this case any differences in the sample material are eliminated, provided the material does not change with prolonged and repeated exposures to elevated temperatures.

(2) Distribution of samples manufactured from the same stock, provided the material may be considered "suitable" in the sense of the above definition.

The complete programme TX 44 is based on the second alternative. All samples of one material were prepared from the same stock. The suitability of the materials is considered proven for the very pure noble metals gold and platinum used in this programme.

Application of method 2 to technical materials offers the additional opportunity for testing the homogeneity or uniformity of the original stock, which determines whether or not the thermophysical data obtained on technical materials may be considered sufficiently representative to justify great accuracy of the measurements.

1.2 Aim of the Project Section "Thermal Expansion up to 1000°C"

The determination of the thermal expansion of solid materials up to 1000°C is considered conventionally as the most simple measurement within the realm of thermophysical property measurements. Customarily such measurements, especially in industrial laboratories, are performed by means of pushrod dilatometers, using both quartz glass and alumina reference systems.

Inasmuch as this type of apparatus does not yield absolute values and the accuracy of data obtained thereby is frequently considered in doubt, it is a special objective of this project section to determine the accuracy of this type of measurements by using the noble metals gold and platinum.

A further objective is to investigate the thermal expansion behaviour of technical materials up to 1000°C, to find out if such materials can be used as calibration standards and to establish if an extension of the study on these materials to higher temperatures is justified.

16 laboratories participated in this programme section. They are listed in Chapter 6.

1.3 Mode of Presentation of the Experimental Results

Thermal expansion measurements involve independent determination of the length change and the temperature of the sample. In most cases the measurements are carried out under conditions of continuously increasing or decreasing temperature and not at a fixed temperature level. For the sake of clarity the representation of all experimental results in the Tables 1-15 was limited to intervals of 1000°C.

The results are shown in diagrams depicting the measured $\Delta l/l_0$-values (%) versus the measured temperature T (°C), following the usual convention. As to comparing the measurements obtained by different authors, it is customary to select fixed measurement temperatures and to compare the discrepancies in the length change values at these fixed temperatures. This procedure has its limitations however, since the discrepancies of the results can also be due to errors in the temperature determination.

In view of the selected arrangement of the diagrams, only the band widths of scatter for the relative length change are continuously connected by straight lines. The average results obtained by each individual participant are in 200° intervals and are depicted outside this band (see Figures 1, 2, 5, 13, 16, 17, 20, 22, 23, 27, 28, 30, 31, 33).

The deviation of the individual mean values from the group average or from the data of a selected reference material is demonstrated as a function of temperature, using the group mean value or the reference data as the zero-line (Figures 3, 4, 8, 9, 10, 11, 12, 14, 18, 21, 24, 29, 32, 36).

A third kind of diagram is used for the analysis of the sources of errors. Following a suggestion of participant 32, the individual mean thermal expansion values of a considered material are plotted against the individual values of another material measured by the same participant under the same conditions. In such a plot, deviations due to systematic errors lead to scatter only in one diagonal direction, whereas scatter in the other diagonal direction can only be caused by non-systematic sources or by differences between the samples (see Figures 7, 15, 19, 25, 26, 34).

2. COOPERATIVE THERMAL EXPANSION MEASUREMENTS FOR DETERMINATIONS OF THE ACCURACY OF DATA OBTAINED BY PUSHROD DILATOMETRY

A pushrod system always measures the difference in length change between the sample and a reference material as incurred during heating and cooling. Thus the length change of the reference material must be known. Accordingly, the method is "non-absolute". As nearly all measurements within this cooperative programme were carried out by means of pushrod dilatometers, an attempt was made to correlate these data with data obtained by other methods and literature data. Pure gold and platinum were selected as suitable materials for this purpose.

2.1 Expansion of Gold and Platinum by X-Ray and Optical Methods

For the stable noble metals gold and platinum so-called "absolute" expansion data can be obtained by using either optical methods (such as twin microscopes or interferometers) or X-ray techniques involving a determination of the lattice expansion. Inasmuch as gold and platinum have a cubic face-centresed lattice structure, thus featuring an isotropic lattice expansion, the two methods can be expected to yield identical results.

In fact, the literature values for the lattice expansion of pure gold (1,2) and platinum (3) obtained by X-ray techniques show good agreement between lattice and bulk expansion values (1,4), the agreement holding for gold up to 900°C and for platinum up to 1100°C. The divergence at higher temperatures is due to thermal generation of lattice vacancies. The lattice vacancies do not influence measurements of the lattice spacing but do affect bulk expansion.

In Tables 1 and 2 the thermal expansion values are compiled for pure gold and platinum. For comparison, lattice expansion values for AGARD gold and AGARD platinum as obtained by participant No.8 and bulk expansion values for AGARD platinum as measured by participant No.32, using twin microscopes, are included as well. The agreement with literature values is good. If average values are computed from all these data, the absolute deviations from the means doe not exceed approximately ± 0.005% provided that the 1000°C values are disregarded for the reasons discussed. Accordingly, the assumption of gold and platinum being suitable materials may be regarded as confirmed.

In Figure 1 and Figure 2, the small scatter of the values of Tables 1 and 2 is shown graphically*. The participants agreed to use the following data as the most probably accurate ones as reference data:

for gold the lattice expansion data due to Merryman and Kempter (Ref. (2))

for platinum the bulk expansion data due to Kirby (Ref. (4))

As these reference data will be used in all further evaluations, the discrepancies between the data compiled in Tables 1 and 2 are illustrated in Figures 3 and 4, using the selected reference data as the zero-line.

2.2 Experimental Results on Gold and Platinum as Obtained by the Individual Participants of the Cooperative Measurement Programme

2.2.1 Characterization of the Noble Metal Sample Materials

The gold samples, supplied by Degussa, West Germany, were fabricated especially for the programme by casting, followed by rolling or drawing down to the required final diameter. No subsequent heat treatment was applied. The purity of this material was 99.999%.

The platinum material, supplied by Heraeus, West Germany, underwent a special fabrication process. The purity of the material was 99.999%. All samples were provided in a more or less cold-worked state, the degree of cold working depending on the sample sizes. Detailed information on the sample material is compiled in Chapter 7.

2.2.2. Pushrod Systems Employed

The pushrod dilatometers employed for the measurements involved quartz glass or alumina reference systems (see Table 3). More details concerning the apparatuses and applied measuring methods are compiled in Chapter 6.

2.2.3 Average Values of the Bulk Expansion Data and their Reproducibility as Obtained by the Individual Participants

Most of the participants presented their results as average values of several runs and added information about the band width of scatter to characterize the reproducibility of their measurements. The quantitative characterization of the band width of scatter was not uniform. Only a few participants used the most exact way for characterizing the band width, i.e. the standard deviation, the reason being that in some cases the number of runs did not seem to justify deriving standard deviations. Therefore, the 2/3-band width or the whole band width of scatter of the single runs were used as well. Some participants did not give any information at all on the band width of scatter obtained by repetitive measurements.

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* For an explanation of the mode of representation see Section 1.3, page 2.

According to the data submitted by the participants, Tables 4 and 5 list the individual average values as well as the individual absolute band widths of scatter. These individual average values should be free of random errors, if a sufficient number of measurements were made under equal conditions. Additonally, Tables 4 and 5 also list the group mean evaluated as arithmetic mean of all individual average values at the temperatures considered.

In some cases the participants described observations regarding the stability of the gold and platinum samples during the tests. Summarizing this information, in general the results of the first run were not taken into consideration for evaluation of the average values, the reason being uncertainty as to possible recrystallization. The subsequent runs showed no changes in thermal expansion behaviour attributable to recrystallization.

2.2.4 The Group Scatter of the Individual Experimental Results

For Figure 5, the previously discussed mode of presentation is applied to the individual mean values of Tables 4 and 5. In Figure 6 the band width of the group scatter is plotted as a function of temperature for both of the investigated metals, gold and platinum. With reference to the work programme described in the introduction, the width of the bands of scatter for gold and platinum as shown in Figures 5 and 6 represents a quantitative characterization of the data spread which must be taken into consideration for measurements of thermal expansion values on samples of equal history obtained by various pushrod techniques, different measurement conditions, and different specialists.

It is apparent that the group scatter of the individual means is significantly larger than the individual reproducibilities, the difference amounting to a factor of 3 to 4.

For both gold and platinum, the band width of the group scatter increases with temperature, the augmentation for gold exceeding that for platinum by a factor of 1.2. If the difference in the expansion values for these two noble metals is taken into consideration, a systematic error entirely due to the temperature measurement would lead to a factor of 1.6.

2.2.5. An Analysis of the Absolute Accuracy of Thermal Expansion Data Obtained by Pushrod Techniques

Before starting the comparative calibration procedure of the individual mean data, using the selected reference data, an attempt was made to decide if the group scatter is effectively caused by systematic errors of the participants only.

In Figure 7 the individual mean values for the thermal expansion of gold at 300°, 500°, 700° and 900°C are plotted versus the corresponding data of platinum, the data referring to each participant who provided data for both gold and platinum. As can be seen, the group scatter of the individual mean values is primarily caused by systematic errors.

The scatter due to non-systematic errors occurring is difficult to analyse to but a minor extent. If it is assumed that the gold and platinum samples used were equal, it must be concluded that the individual averaging was not sufficient to eliminate *all* random errors or that the data used for the plot (gold and platinum) were measured under different conditions

Disregarding this small uncertainty, the individual systematic error of the participants can be eliminated by referring the submitted individual means to the previously selected reference data for gold and platinum (Tables 1 and 2). This is done in Figures 8 and 9, using the reference data as the zero-line.

These deviations shown may be considered as correction terms to be applied to the results of any individual apparatus involved, thus permitting each participant to obtain absolute values, with an accuracy not better than that given in Tables 1 and 2 for selecting the reference data (± 0.005%), provided that the correction is not dependent on the magnitude of the expansion to be corrected. A comparison of the individual correction terms for the various participants indicates as shown in Figure 10 that this condition is satisfied to but a limited extent.

As shown later, the use of these correction terms has brought about in some cases only a considerable reduction of the group scatter.

An analysis of the group results seems to be much more important than the given separate correction of individual mean values. If an average of the individual means is computed, it is found, as shown in Figures 11 and 12, that these group averages are surprisingly close to the selected reference data, the deviation from the latter for gold being even smaller then the accuracy for selecting the reference data.

This indicates that by means of a group effort involving not more than nine, effectively ten* independent participants, it is possible to derive absolute values with sufficient statistical certainty. The reason lies obviously in the fact that the systematic errors of various apparatuses follow an approximately symmetrical distribution.

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* Values of the participants 8, using two different apparatuses, are considered as supplied from two independent sources (indicated in the Figures as No.8 and No.2).

The band widths shown in Figures 11 and 12 represent the group scatter of the individual means about the group mean.

It can be expected that additional new participants should improve the accuracy obtainable by this procedure.

2.3 Conclusions Regarding the Accuracy of Pushrod Dilatometer Measurements

The cooperative measurements on highly pure gold and platinum by nine, effectively ten, participants permit to draw the following conclusions:

(1) The *accuracy* of the so-called "absolute" thermal expansion measurements, using optical or X-ray methods, expressed as length change $\Delta l/l_0$ in percent or as lattice expansion $\Delta a/a_0$ amounts to ± 0.005% at 900°C. This accuracy is approximately equivalent to a relative deviation of 0.3% from the mean thermal expansion value.

(2) The individual *reproducibility* of pushrod dilatometer measurements is found to be better than ± 0.010% expressed as length change $\Delta l/l_0$ throughout the entire temperature range investigated (300° – 1000°C), although this value differs somewhat between the various participants and depends slightly but not systematically on the temperature region.

(3) The *individual systematic error* of the participants causes a group scatter of the individual mean values, the band width growing

from ± 0.015% at 300°C up to
± 0.025% at 900°C.

These values are expressed again in length change $\Delta l/l_0$.

(4) The systematic error of the individual mean values is of the same magnitude for gold and for platinum, showing that inaccuracy of the temperature measurements cannot be the *main source of the systematic errors* in pushrod measurements. A similar conclusion can be drawn by comparison of the systematic errors using silica glass or alumina reference pushrod systems. No fundamental differences could be found between the results obtained with the two different oxide reference systems either in terms of systematic errors or in the individual reproducibility.

(5) Averaging of the individual mean value leads to mean values of the cooperating group very closely approximating to the assumed true values of the thermal expansion of the considered material, the absolute deviation not exceeding approximately ± 0.005%. This leads to the highly important finding that the individual systematic errors of the participants have approximately a symmetrical distribution, thus causing the group mean values to closely approximate the true values, the deviation being no greater than that of an independent absolute measurement. This finding is based on a group of ten independent individual mean values. The result seems to offer an opportunity to obtain true values of thermal expansion up to 1000°C by a group of independent pushrod systems without calibration, merely on a statistical basis.

(6) Provided that all individual random errors are eleiminated by a sufficiently large number of repetitive experiments, an individual calibration of pushrod results should lead to an individual absolute accuracy which depends merely on the uncertainty of the data for the selected reference material. In view of the above conclusion elaborated in point 5, the calibration does not seem to lead to a higher accuracy than the statistical evaluation of a group effort. In other words, for both cases an absolute accuracy better than ± 0.005% expansion cannot be attained.

3. COOPERATIVE THERMAL EXPANSION MEASUREMENTS ON TECHNICAL MATERIALS USING PUSHROD DILATOMETRY

3.1 Derivation of True Data from a Statistical Evaluation of a Group Effort

The aim of Chapter 3 is to represent the thermal expansion results obtained on technical materials in the cooperative measurement programme employing pushrod dilatometer techniques. The results formulated in Section 2.3 can be used as a basis for appraising the measurements on the technical materials. If approximately ten independent participants measure the thermal expansion of a technical material by pushrod techniques and if the resulting group scatter of the individual mean values is not greater than that obtained on gold or platinum, then the following conclusions can be drawn:

(1) Samples manufactured from one charge of the materials involved exhibit a thermal expansion behaviour as uniform and stable as that for the gold and platinum samples used in this programme. No additional uncertainty arose due to using a technical material instead of a noble material. Such materials can be considered suitable in the sense of the definition given in Section 1.1.

(2) The group means of the results obtained on the technical materials at any considered temperature can be expected to represent the absolute true thermal expansion of the material considered, the accuracy amounting to ± 0.005% expansion.

If the width of the group scatter on a technical material should be found larger than in the case of gold or platinum, then differences are likely to exist between the samples distributed.

The certainty of these conclusions is limited if the number of participants is too low. This situation applies to a few of the sample materials investigated in this programme. In such cases it is necessary to eliminate the individual systematic errors by calibration work using a suitable standard so that the true expansion is obtained individually and not by a group effort.

Based on the given conceptions, the thermal expansion data obtained for the austenitic alloy, alumina, Ta/10 W, tungsten, three different types of graphite, copper, and silica glass are discussed in the following sections.

3.2 Experimental Results on Technical Materials Presently Used in the Cooperative Programme

3.2.1. Technical Materials for High Temperatures with Thermal Expansion Coefficients Exceeding 15 x 10^{-6} ($°C^{-1}$)

Cooperative measurements on a material of such a high coefficient of thermal expansion are of particular importance since temperature errors alter the result of the measurement decisively. In addition, a direct comparison with the selected reference material gold (α_m between 15 and 17 x 10^{-6} ($°C^{-1}$)) facilitates the appraisal as to whether or not the measurement techniques are suitable. A Nb-stabilized 16Cr2ONi austenitic steel was selected because of its proven thermal stability up to 1000°C. The mean thermal expansion coefficient of this material lies between 17.5 and 20 x 10^{-6} ($°C^{-1}$).

The results can be considered as representative for thermally stable high temperature iron metal alloys.

3.2.1.1. Characterization of the austenitic alloy

The Nb-stabilized 16Cr2ONi steel had the following composition:

0.009% C	0.01 % Mo
1.2 % Mn	16.41 % Cr
0.27 % Si	0.10 % Nb
0.005% P	0.009% N
0.006% S	the remainder being iron.
19.90 % Ni	

The material supplied by the Deutsche Edelstahlwerke, Krefeld, West Germany, was made especially for this programme. Further details will be given in Chapter 7.

3.2.1.2. Experimental results of the participants as obtained for the austenitic alloy

Thermal expansion measurements on samples of the austenitic alloy were carried out by eight participants (as compared to nine, respectively ten, for gold and platinum). Accordingly, the condition of a sufficient number of different laboratories participating may be considered satisfied. The individual results are compiled in Table 6 and depicted in Figure 13. The group scatter was found to be surprisingly small. Figure 14 illustrates a comparison of the group scatter for gold and the austenitic alloy, using the corresponding group means as the zero line. As can be seen, in the case of the austenitic alloy the band width of scatter does not exceed ± 0.02% expansion and is definitely smaller than that of gold.

Supplementing the conclusions described in Section 2.3 of the report, the following inferences can be formulated:

(a) As far as thermal expansion behaviour is concerned the austenitic alloy used in the Cooperative Measurement Programme is homogeneous and stable. It can serve as a suitable calibration material for thermal expansion measurements.

(b) As the conditions for a statistical evaluation are satisfied in spite of not more than seven laboratories participating, the group mean obtained should represent the true value of thermal expansion with an absolute accuracy better than ± 0.005% expansion.

3.2.1.3. Analysis of the group errors

Since the group scatter obtained for the austenitic alloy was found to be lower than that obtained for gold, a further reduction of the band width of scatter by calibration could not be expected. In spite of this, using the previously discussed error analysis plot (here austenitic alloy versus gold), in Figure 15 an attempt was made to find pointers as to the nature of the deviations involved. Apart from the data referring to the 300°C region, the non-systematic errors seem to be more pronounced.

Consequently, a calibration procedure aimed at eliminating individual systematic errors will not lead to absolute individual values of greater accuracy than those derived from the previously discussed statistical evaluation of the group efforts (see Section 3.2.1.2.). To prove this, Figure 16 shows results of such a procedure, indicating that the band width of the group scatter after calibration is greater than before it (compare Figure 13).

3.2.2. Alumina as a Technical Material with an Intermediate Expansion Coefficient of about 8×10^{-6} ($°C^{-1}$)

For the intermediate range of expansion coefficients the highly refractory alumina was selected as it seems to be suitable for measurements even above 1000°C dealt with in the parallel cooperative project section. In addition, a direct comparison with the selected reference material platinum (average expansion coefficient α_m = 9-10.5 x 10^{-6} ($°C^{-1}$)) appears favourable.

3.2.2.1. Characterization of the alumina tested

The alumina samples were supplied by Degussa, Frankfurt, West Germany. All samples were produced especially for this programme and great care was taken to ascertain equal process conditions for all specimens. The final material had a purity of 99.5% Al_2O_3 and a density of 3.8 g/cc. More details on the material will be given in Section7.

3.2.2.2. Experimental results

Measurements on Al_2O_3 samples were carried out by nine participants. The individual means from each laboratory are listed in Table 7. Figure 17 illustrates the group scatter of the results and Figure 18 the deviation of the individual means from the group average as compared to the analogous parameter for platinum.

At the first view, the deviation appears to very similar to that of platinum although there is some indication of irregularities. In addition, as also shown in Figure 18, the group mean of Al_2O_3 results used as the zero line does not concur with the literature data for Al_2O_3 as given in the Handbook of Thermophysical Properties (5). Therefore, an analysis of the group errors appeared to be absolutely imperative.

3.2.2.3. Analysis of the group errors of alumina

An error analysis plot was constructed using the individual means of alumina versus those of platinum obtained by the same participant with the same apparatus.

As can be seen from Figure 19, strong indications for systematic errors exist. The difference of this finding as compared to the results for the case of the austenitic alloy is probably due to the fact that the groups of contributing participants were rather different. To give an example, participant No.12 has contributed only data for Al_2O_3 and platinum and does not appear in any group concerned with other materials.

3.2.2.4. Correction procedure based on a calibration with platinum

To eliminate the systematic errors, a calibration of the individual apparatuses with platinum seemed useful. The result is shown in Figure 20. Comparison with the results obtained prior to calibration (Fig.17) shows that the group scatter was significantly reduced by the calibration. This permits the conclusion that the original deviations were indeed primarily due to systematic errors. In Figure 21 the deviations of the individual means after calibration are compared to the previously obtained platinum results, the zero-line for Al_2O_3 now being represented by the new group means resulting after calibration. The original group means are included for comparison.

Figure 21 permits three important conclusions:

(1) The repeatedly discussed finding that the group mean leads to the true expansion value of a given material with an accuracy comparable to that of individual results by so-called "absolute" methods demands that there should be no difference between the group means before and after calibration. Actually, the observed deviation of 0.001% expansion at 300°C and 0.005% at 900°C may be construed as confirming the above claim in view of the limited accuracy of independent absolute measurement as given in Table 2 in Chapter 2.1 showing values of 0.001% at 400°C and 0.0065% at 900°C.

(2) It is seen that the true expansion of the special Al_2O_3 used in this programme still differs from the cited literature data, which were deduced by averaging a great number of results obtained for different more or less characterized materials.

This justifies the proposition that the group averages on the thermal expansion behaviour of sintered Al_2O_3 here obtained should be considered preferable to the literature data for alumina accepted to date (Ref.(5), page 41).

(3) Concerning the reduced band width of the group scatter after calibration, a statement can be made as to the accuracy of the true expansion data obtainable by one individual pushrod mean result after calibrating with the selected reference material. This accuracy is not worse than ± 0.01% expansion, but probably not better than the repeatedly discussed accuracy of the group means (0.005%).

Summarizing all results here reported for sintered alumina and especially considering the extremely small group scatter obtained after calibration, the material employed can be regarded highly suitable as a standard. Furthermore, in view of the small thermal conductivity of alumina and the correspondingly slow attainment of thermal equilibrium during heating and cooling, the small group scatter may be construed as indicating that a small thermal conductivity does not prevent accuracy of the thermal expansion data obtained with pushrod dilatometers. Thus the alumina material must be strongly recommended as a sample material for the second project section concerned with thermal expansion measurements above 1000°C.

3.2.3. Refractory Metals with Average Thermal Expansion Coefficients Between 4.5 and 7 x 10^{-6} ($°C^{-1}$)

For this group of materials a vacuum-cast Ta/10 W alloy and a sintered pure tungsten were selected for study. Both materials are considered as potential candidates as high temperature standards.

3.2.3.1. Characterization of Ta/10 W and tungsten sample material

The Ta/10 W alloy was fabricated by Fansteel Metals Corporation, Baltimore, USA, and distributed by the US Air Force Materials Laboratory, Dayton, Ohio. This material was a special order for this programme to ascertain all samples being made from one melt. The same type of refractory alloy was studied intensively within a research programme of the US Air Force Materials Laboratory. The composition of this alloy is claimed to be:

W	9.11 %	Nb	0.02%	C	19 ppm
Cr	0.002%	Zr	0.01%	O	20 ppm
Fe	0.01 %		balance Tα		

The sintered tungsten was specially prepared by the Metallwerk Plansee, Reutte, Austria, for this programme, using a special pure raw material. The density of the dilatometer samples reached after swaging and subsequent heat treatment was 100%. The purity is reported by the manufacturer to be 99.98%. More details of both materials are given in Chapter 7.

3.2.3.2. Experimental results of the participants as obtained for the refractory metals

Four participants have contributed data for the Ta/10 W alloy, seven participants for tungsten. The results are compiled in Tables 8 and 9, and shown graphically in Figures 22 and 23. In both cases, the group scatter is larger than for the previous results.

The deviations from the group means as compared with platinum are shown in Figure 24, exhibiting a worse group scatter than for the noble metal. Only for tungsten the group of investigators was numerically comparable to that for platinum.

For the case of Ta/10 W it is evident from Figure 22 that the data for one of the only four contributors lie far outside the scatter band for the three others. For this reason, a second group scatter band is shown for Ta/10 W in Figure 24, for which the greatly deviating results were disregarded. In this case the group scatter (only three participants) is seen to be diminished to ± 0.010% expansion which is significantly less than was found for platinum.

For these three participants used for the smaller group scatter band of Ta/10 W, measurements were also available for the reference material platinum so that an error analysis plot could be constructed as shown in Figure 25. The small deviation of the results between those three participants cannot be interpreted as systematic. Therefore no improvement could be obtained by correcting the individual mean data on the basis of a calibration of the apparatuses with platinum.

Disregarding the small number of participants, the Ta/10 W alloy appears to be a suitable material for a group effort involving thermal expansion measurements, and it is hoped that the results of the experiments above 1000°C will confirm this prediction.

As to the tungsten samples, an error analysis plot (tungsten versus platinum) as shown in Figure 26 is interpreted as indicating an influence of both systematic and non-systematic errors. Consequently, corrections of the individual mean by using platinum as reference material leads to but a small improvement of the group scatter as illustrated in Figure 27. This procedure shifted the group means by approximately 0.007% expansion at 500°C to 0.021% expansion at 900°C.

All these findings indicate that a further source of error must be the reason for these uncertainties with the sintered tungsten samples, especially in view of the results for the previously discussed materials as obtained by the same participants with the same apparatuses using the same measurement procedures. This leads to the assumption that some of the sintered tungsten samples may have been different from the others or that contamination occurred during the measurements. Without further control of these variations, this sample material failed to prove suitable in the sense of the previous definition.

3.2.4. Graphites as Materials with Extremely High Refractoriness and very Low Average Expansion Coefficients (Down to 2 x 10^{-6} ($°C^{-1}$))

In view of the fact that polycrystalline graphite consists of a multitude of highly anisotropic grains and crystallites and considering that its bulk expansion is smaller than that of a graphite single crystal (on account of fissures taking up a significant portion of the expansion of the individual crystallites), it was highly surprising that in a report by Gaal (6) polycrystalline graphite was claimed to be a suitable standard for high temperature thermal expansion measurements. Based on the recommendations of the cited report, three grades of polycrystalline fine-grain graphites were selected for investigations within the framework of this cooperative programme.

3.2.4.1. Characterization of the measured graphites

(1) POCO graphite AXM-5Q, produced by POCO Incorporation, Garland, Texas, and donated by the US Air Force Materials Laboratory in Dayton, Ohio. This material is an ultra-fine-grain graphite manufactured by a proprietory process and has a bulk density between 1.70 and 1.78 g/cc without showing any significant bulk anisotropy in physical and mechanical properties.

(2) AAQ1 graphite produced and donated by the Los Alamos Scientific Laboratory, Los Alamos, New Mexico. This material is an extruded resin-bonded graphite of extreme uniformity, having the relatively high density of 1.90 g/cc. The samples from this material were cut exclusively with their greatest dimension parallel to the direction of extrusion because of the limited diameter of the rod stock supplied.

(3) RVD graphite, produced and donated by Union Carbide Corporation, New York, N.Y. This material is a molded graphite having a bulk density of approximately 1.90 g/cc. The samples were cut from the same stock which had been used for a US Air Force Programme, carried out by Arthur D.Little Inc. (7). Samples were cut from the graphite billets with their greatest dimension both with and across the molding direction.

3.2.4.2. Experimental results on the graphites

All graphite types were measured by five or six participants respectively. The results of all measurements are compiled in Tables 10-13.

As the first mentioned types AXM-5Q and AAQ1 were measured only in one direction, the individual means and the band of group scatter are shown graphically together in one plot (Fig.28).

The excellent agreement of the individual means obtained with pushrod systems can be easily recognized. The data due to participant 32 are shown in parentheses for comparison. These data were taken from the cooperative high temperature (> 1000°C) expansion programme carried out by Westinghouse (Ref.6)* and were not used here for the statistical evaluation. The group scatter of the original results without correction with a reference material as shown in Figure 29 does not exceed ± 0.020% expansion at 900°C and corresponds very closely to the scatter obtained for platinum. Accordingly, those two types of graphites are equally suitable reference materials. In addition, the repeatedly discussed conclusions regarding the derivation of true values by a statistical evaluation of a group effort are perfectly applicable to these graphites without reservation.

The results concerning the type RVD for the different directions are shown in Figures 30 and 31.

The group scatter for both directions is comparable to those of the other graphites tested. In view of the much coarser grains of RVD, this result, shown in detail in Figure 32, is highly surprising. It indicates great uniformity of all graphite samples studied.

All three types of graphites investigated can be recommended as reference materials for high temperature thermal expansion measurements.

3.3 Conclusions Concerning the Suitability of Technical Materials for the Cooperative Programme TX44

The cooperative measurements of thermal expansion on the selected technical materials austenitic alloy, alumina, Ta/10 W, tungsten and three types of graphite permit to draw the following conclusions regarding their suitability in the sense given in Section 1.1.

(1) *Austenitic Alloy*

Eight participants have contributed results for this material. Therefore the conditions for a statistical evaluation are satisfied. It was found that the established group scatter for the austenitic alloy was smaller than that for gold. A comparison with the values obtained by absolute measurement techniques allows the conclusion that the group mean can be considered as the true value of thermal expansion with an uncertainty of ± 0.005% expansion. It was found by an error analysis that the group scatter of austenitic alloy is caused merely by *non-systematic errors.*

In order to eliminate the individual systematic errors, a calibration procedure using gold as a reference material does not lead to absolute individual values with a greater accuracy than those values derived from a statistical evaluation of a group effort. The austenitic alloy was found to be suitable in the sense described above and can serve as a useful calibration material for thermal expansion measurements.

(2) *Alumina*

Nine laboratories have participated in the measurements of alumina. The obtained group scatter is smaller than that found on platinum. Therefore the group mean can be regarded as being the true value for thermal expansion with an accuracy of ± 0.005% expansion.

By the error analysis plot alumina versus platinum it was found that the group scatter is merely caused by *systematic errors.* A calibration procedure using platinum as reference material led to a reduction of the group scatter of 50%.

Alumina can be recommended as a highly suitable calibration material for thermal expansion measurements as well as a candidate material for the second project section of the programme TX44 (thermal expansion above 1000°C).

(3) *Ta/10 W*

The group of four cooperating laboratories does not justify a statistical evaluation. The results of three laboratories are in good agreement. In spite of the disagreement of the fourth laboratory the suitability of this material must be clarified in the second project section by additional contributing laboratories.

(4) *Tungsten*

Seven laboratories were engaged in the measurements of the sintered tungsten material. The group scatter of the individual means was 0.120% expansion at 900°C (Pt for comparison: 0.040% expansion). The broad scatter in this special case may have been caused by differing or contaminated samples. Therefore a suitability in the sense given in Chapter 1.1 could not be proved so far for the sintered tungsten material.

(5) *Graphites*

All graphite types were measured by five or six participants, respectively. The group scatter for each type is not greater than for platinum. In particular the ultra fine-grain AXM-5Q-type shows the very low group scatter of only ± 0.010% expansion up to 700°C. In view of the good agreements of the individual results, the group mean for the graphite types investigated may be considered as the true values of thermal expansion with an uncertainty of ± 0.005% expansion, although the conditions for a statistical evaluation, as stated above, are not satisfied.

The graphite types AXM-5Q, RVD, and AAQ1 are suitable materials in the sense of Section 1.1 and can be recommended as reference materials for thermal expansion measurements.

4. MEASUREMENTS ON MATERIALS WITH EXTREMELY LARGE AND EXTREMELY SMALL COEFFICIENTS OF THERMAL EXPANSION

Pure copper is known to have an extremely high coefficient of thermal expansion amounting to 20×10^{-6} ($°C^{-1}$) and silica glass has one of the lowest values of solids not exceeding 0.55×10^{-6} ($°C^{-6}$). Both of these materials were selected for study. Regarding copper, it was hoped that if suitable it might prove acceptable as a reference material instead of the noble metals. In the case of silica glass, its use as the material for the reference system in most push-rod dilatometers warranted an investigation as to its stability with respect to thermal treatment.

4.1 Characterization of the Materials

The copper samples were manufactured by the American Smelting and Refining Company, New York, N.Y., and purchased as one lot to ensure fabrication from one melt. The material was claimed by the manufacturer to have a purity of 99.999%. For more information see Chapter 7.

The silica glass was produced by Corning Glass, Corning, N.Y., and specially heat treated. It was donated by Westinghouse Astronuclear Laboratory, Pittsburgh. For more information see Chapter 7.

4.2 Experimental Results on Copper

The copper was studied by seven participants. The results are compiled in Table 14 and plotted in Figure 33. As can be seen, the group scatter is surprisingly great.

Since five participants had also studied the reference material gold, a corresponding error analysis plot could be constructed as depicted in Figure 34. Although there are indications of systematic errors, a superposition of non-systematic errors is evident. As could be expected, a calibration with the reference material gold led to but a minor reduction of the group scatter. Significant uncertainties remain and it is, therefore, concluded that the copper tested is no suitable replacement for gold as a reference material, and even less so for the austenitic alloy. Both gold and the austenitic alloy exhibit high expansion coefficients similar to copper.

4.3 Experimental Results on Silica Glass

Silica glass was studied by ten participants. The results are compiled in Table 15 and depicted in Figure 35 where a distinction is made between pushrod dilatometers with silica glass and alumina reference systems (dotted lines). Considering that the expansion of the alumina reference system exceeds that of the silica glass sample by a factor of 15, the agreement of the results obtained with the two types of systems may be considered surprisingly good.

The group scatter of all individual means for the silica glass samples are comparable with that for gold as illustrated in Figure 36 depicting the deviations from the group averages. From this figure it is concluded that the group scatter is not affected by the absolute magnitude of thermal expansion of the sample. Gold having an expansion coefficient of 15 to 17 x 10^{-6} ($°C^{-1}$) yields the same scatter as silica glass having 0.5 x 10^{-6} ($°C^{-1}$). If the systematic errors were caused by temperature measurements, then the group scatter should be strongly dependent upon the absolute magnitude of the thermal expansion. Considering that this is not observed, errors in temperature measurement should not be the cause for the actual scatter.

As can be seen in Figure 35, the data of participant No.21 lie considerably outside the region encompassed by the other measurements and it is noteworthy that these strongly differing data appear to involve an error in the zero point. If, therefore, these data are disregarded, an entirely different band for the group scatter is obtained as shown in Figure 36. This band has an extremely small width in the region of low temperatures. This indicates that in the low and intermediate temperature range the scatter of the individual averages is indeed affected by systematic temperature errors but this effect disappears completely at higher temperatures (900°C).

Regarding the absolute expansion value of the quartz glass samples studied, a comparison can be made between the group mean representing the true value and the individual value supplied by participant No.42 obtained by twin microscopes and claimed as absolute. This deviation increases from 0.006% expansion at 300°C up to 0.012% at 900°C. Again this deviation falls into the accuracy band claimed for so-called absolute measurements.

By the definition of suitability used in this report, quartz glass exhibits exactly the same qualities as the materials shown to be good, i.e. gold, platinum, the austenitic alloy, alumina and ultra-fine-grain graphite.

According to all findings, there is no reason to reject silica glass reference systems in pushrod dilatometers, at least up to 900°C. On the other hand, there are some indications that other materials with a higher expansion coefficient could be used with equal success in the same application.

4.4 Conclusions Concerning the Suitability of Copper and Silica Glass as Sample Materials

Copper and silica glass were considered as potential candidate materials because of their extremely differing expansion behaviour (20 x 10^{-6} (°C) and 0.5 x 10^{-6} (°C)). The experimental results led to the following conclusions on their suitability:

(1) *Copper*

Seven participants contributed results on highly pure copper. The resulting group scatter for copper is approximately three times larger than that for gold. As this group scatter was found to be caused by non-systematic deviations, it could not be reduced significantly by calibration with gold. It is assumed that copper can be considered as being extremely sensitive to contaminations and as unstable because of recrystallization. The used copper cannot be recommended as a suitable material for thermal expansion measurements, especially the highly pure copper cannot replace gold as a reference material for calibration procedures.

(2) *Silica Glass*

Ten laboratories have participated in the measurements of silica glass. The group scatter obtained is ± 0.025% expansion at 900°C, comparable with that for gold. At 300°C the group scatter is decreased to only one-third, if the deviating results of one participant in ten are disregarded. The group mean can be considered as the true value of the thermal expansion with an accuracy of ± 0.005% expansion.

This group mean is in agreement, within the error limit, with the results of one participant, obtained by an absolute method (twin microscopes).

According to these facts, silica glass can be considered as a suitable material for thermal expansion measurements when a very low expansion behaviour is desired.

5. SUMMARY

This report represents a compilation and evaluation of thermal expansion measurements to 1000°C by means of pushrod dilatometers as carried out by a group of cooperating laboratories. Samples having the same history were used for all tests. The materials investigated were gold, platinum, an austenitic alloy, a Ta/10 W alloy, tungsten, alumina, three types of graphite, copper and silica glass.

From the mean values of the individual contributors, band widths of group scatter as well as group averages were derived. In addition, error analyses were carried out to show if the errors involved were predominantly of a systematic or of a non-systematic nature.

For gold and platinum, the absolute accuracy of the group averages amounting to 0.005% expansion was found to be no worse than that of individual averages by X-ray and optical measurements. This led to the conclusion that for a statistical evaluation of a group effort, seven to ten independent contributors are sufficient to ascertain that systematic errors are distributed symmetrically.

Of the technical materials investigated, the austenitic alloy, the Ta/10 W alloy, alumina, and a special ultra-fine-grain graphite were found to be very suitable reference materials. Tungsten looks promising but requires further studies.

In the case of alumina, a correction of the originally submitted individual averages by calibrating the apparatuses with platinum was found to decrease the band width of group scatter without significantly changing the group averages, thus indicating that the original deviations were primarily due to systematic errors. For all other materials, the calibration procedure did not reduce the band width of group scatter to any significant extent but again the group averages remained unchanged.

A comparison of the accuracies obtained on materials with very high and very low expansion coefficients has shown that in the temperature range around 900°C the systematic errors are certainly not caused by errors in temperature measurement but the latter seem significant in the intermediate and low temperature regions.

Regarding the accuracy obtained by the pushrod dilatometers used in this programme it was observed that it is possible for an individual calibration to be less reliable than a statistical evaluation of a group effort and therefore a group effort is preferrable.

For the pushrod dilatometer reference system, up to 900°C silica glass was found to be acceptable but not better than alumina. Ultra-fine-grain graphite looks promising for the same application.

From the group averages obtained, the following mean coefficients of thermal expansion between room temperature and the temperature indicated were found (10^{-6} ($°C^{-1}$)):

	300°C	500°C	700°C	900°C
Gold	14.90	15.50	16.13	16.82
Platinum	9.32	9.66	9.97	10.24
Austenitic Alloy	17.38	18.50	19.15	19.64
Ta/10 W	6.17	6.31	6.50	6.70
Alumina	6.78	7.38	7.88	8.19
AXM-5Q	7.08	7.46	7.65	7.89
AAQ1	2.03	2.40	2.59	2.80
RVD (with grain)	3.54	3.90	4.09	4.27
RVD (across grain)	3.36	3.63	3.80	4.08

6. PARTICIPANTS OF THE PROJECT SECTION, DEALT WITH IN THIS REPORT

6.1 Participants from France

G.Sertour
Sud-Aviation, Groupe Technique de Paris, Laboratoire Central,
55 rue Victor Hugo, 92 Courbevoie, France.

Apparatus:	Pushrod dilatometer with an Al_2O_3-system
Heating rate:	5°C/min
Atmosphere:	Air, 10^{-5} torr
Type of temperature measurements:	Thermocouple
Sample size:	5ϕ x 30 mm

6.2 Participants from Germany

F.Bollenrath
Rhein.-Westfäl. Techn. Hochschule Aachen,
Institut für Werkstoffkunde, 51 Aachen, Germany.

Apparatus:	Pushrod dilatometer with a quartz system
Heating rate:	0.5°C/min (200°C) to 1.5°C/min (1000°C)
Atmosphere:	Air (Au, Pt); air 10^{-5} torr (Al_2O_3, Cu, W, Ta/10 W aust. alloy, graphites)
Type of temperature measurements:	Pt-Pt/Rh thermocouple
Sample size:	3 x 3 mm cross section, 40 mm long

K.Bungardt, W.Spyra, K.Wetzlaer
Deutsche Edelstahlwerke AG,
415 Krefeld, Germany. Oberschlesienstrasse 16

Apparatus:	Pushrod dilatometer with a quartz system
Heating rate:	1°C/min (200°C), 3°C/min (above 200°C)
Atmosphere:	Argon, 680 torr
Type of temperature measurements:	Palladium-Platinum thermocouple
Sample size:	4 ϕ x 50 mm

E.Fitzer, S.Weisenburger
Universität Karlsruhe, Institut für Chemische Technik,
75 Karlsruhe, Germany. 12 Kaiserstrasse

Apparatus:	Pushrod dilatometer with a quartz system
Heating rate:	2°C/min
Atmosphere:	Air (Au, Pt, Al_2O_3), argon (Cu, Ta/10 W, aust. alloy, W, graphites)
Type of temperature measurements:	Pt-Pt/Rh thermocouple
Sample size:	5ϕ x 50 mm

E.Gugel, E.Kupzog
Bak Beratungs- und Arbeitsstab
Keramik-Forschungsinstitut, 8633 Oeslau bei Coburg, Germany. Postfach 44

Apparatus:	Pushrod dilatometer with an Al_2O_3-system
Heating rate:	5°C/min
Atmosphere:	Air (Au, Pt, Al_2O_3), argon (Cu, aust. alloy)
Type of temperature measurements:	Pt-Pt/Rh thermocouple
Sample size:	4-5° x 50 mm

H.Lehman, P.Thorman
Techn. Universität Clausthal, Institut für Steine und Erden,
3392 Clausthal-Zellerfeld, Germany. Zehntnerstrasse 2A

Apparatus:	Pushrod dilatometer with a quartz system
Heating rate:	1.5°C/min
Atmosphere:	Air
Type of temperature measurements:	Pt-Pt/Rh thermocouple
Sample size:	6ϕ x 25 mm

A.Rabenau, E.Roeder

Philips Forschungs laboratorium GmbH
51 Aachen, Germany, Weisshausstrasse

Apparatus:	Double differential pushrod dilatometer with an Al_2O_3-system
Heating rate:	1°C/min
Atmosphere:	Purified argon
Type of temperature measurements:	Pt 90 Pt/10Rh thermocouple
Sample size:	5ϕ x 40 mm

J.Semmler

Sigri-Elektrographit GmbH
8901 Meitingen, Germany.

Apparatus:	Pushrod dilatometer with a graphite system
Heating rate:	2°C/min
Atmosphere:	Argon
Type of temperature measurements:	Thermocouple
Sample size:	9ϕ x 50 mm

W.Delle

Institut für Reaktorwerkstoffe, Kernforschungsanlage Jülich des Landes,
Nordrhein-Westfalen e.V., 517 Jülich, Germany. Postfach 365

Apparatus:	Pushrod dilatometer with a quartz system
Heating rate:	1.2°C/min
Atmosphere:	Argon
Type of temperature measurements:	Pt-Pt/Rh thermocouple
Sample size:	3.3ϕ x 30 mm

G.Ondracek, G.Hoffman

Institut für Material- und Festkörperforschung, Kernforschungszentrum Karlsruhe,
75 Karlsruhe, Germany. Postfach 3640

Apparatus:	Pushrod dilatometer with a Al_2O_3-system
Heating rate:	2°C/min
Atmosphere:	H_2
Type of temperature measurements:	Thermocouple
Sample size:	5ϕ x 10.6 mm

6.3 Participants from Italy

P.M.Strocchi, V.Lupnc

Laboratorio par la Tecnologia dei Materiali metallici non tradizionali del CNR,
Via Induno 10, 20092 Cinisello, Milano, Italien.

Apparatus:	Pushrod dilatometer with a quartz system
Heating rate:	0.8°C/min (500°C) to 1.5°C/min (800°C)
Atmosphere:	Air (10^{-5} torr)
Type of temperature measurements:	Pt-Pt/Rh or chromel-Alumel thermocouple
Sample size:	4ϕ x 20 mm

6.4. Participants from USA

C.R.Cunnington

Lockheed, Palo Alto Research Lab., Therophysics,
Aerospace Sciences Lab., 3251 Hanover Street, Palo Alto, California 94 304, USA.

Apparatus:	Pushrod dilatometer with a quartz system
Heating rate:	1°C/min
Atmosphere:	Argon
Type of temperature measurements:	Pt-Pt/Rh thermocouple
Sample size:	5ϕ x 50 mm

M.L.Minges, G.L.Denman

Air Force Mat. Lab., Thermal Protective Systems, Space and Miss. Syst. Supp. Branch,
Mat. Supp. Div., Wright-Patterson Air Force Base, Ohio 45433, USA.

Apparatus:	Pushrod dilatometer with a quartz system
Heating rate:	1-2°C/min
Atmosphere:	Purified argon
Type of temperature measurements:	Pt-Pt/Rh thermocouple
Sample sizes:	5ϕ x 50 mm, 4ϕ x 25 mm (Ta/10 W)

R.K.Kirby

Nat. Bureau of Standards,Inorganic Materials,
Washington D.C. 20234, USA.

Apparatus:	Measurement of the length change by twin microscopes
Heating rate:	Measurement at fixed temperatures
Atmosphere:	–
Type of temperature measurements:	Pyrometer
Sample size:	6ϕ x 120 mm

P.S.Gaal

Westinghouse, Astronuclear Lab.
P.O.Box 10864, Pittsburgh, Pennsylvania 15236, USA.

Apparatus:	Measurement of the length change by twin microscopes
Heating rate:	Readings at fixed temperatures
Atmosphere:	–
Type of temperature measurements:	Thermocouple or pyrometrically
Sample size:	5ϕ x 50 mm

J.C.Rowley

Los Alamos Scient. Lab.,
P.O.Box 1663, Los Alamos, N.Mexico 87544, USA.

Apparatus:	Twin microscope, optical extensometer
Heating rate:	Readings at fixed temperatures
Atmosphere:	Argon
Type of temperature measurements:	Chromel-alumel thermocouple
Sample size:	4.7ϕ x 100 mm

7. DETAILS OF THE SAMPLE CHARACTERIZATION

7.1 Gold

Manufacturing Process:

The purification of the marketable "good delivery" bullions of 99.6% was secured by the Wohlwill Electrolytic Refining process.

By this process gold is desposited as crystals of 99.99% on fine gold strips. The gold crystals were remelted and poured into bullions of 12.5 kg.

The next step of refining to 99.999% may be realized by several processes, mostly used is the liquid-liquid extraction method.

As to machining of the sample, gold 99.999% was remelted in a graphite crucible in a high frequency induction furnace and a vacuum better than 10^{-3} torr. The melt was poured into a slit graphite mold of respectively 18 or 30 or 60 mm interior ϕ. The ingots were cold-rolled to rolling sections of 12 to 6 mm ϕ and then cold-drawn through hard metal dies to a final diameter of 3 to 10 mm.

No intermediate annealing was applied because 95% reduction work-hardens gold 99.999% only from Vickers hardness 25 to about 70 kg/mm^2. Finally the samples were cleaned in hydrochloric acid. In this work-hardened state the samples were sent to the research laboratories.

Properties:

Density (g/cm³)	19.29
Grain size	Differs very much with the percentage of reduction, i.e. a rod of 5 mm ϕ with a reduction of 93% has a complete fibre structure, whereas a rod of 10 mm ϕ with a reduction of 70% has still a typical coarse though stretched grain which has a width of some 0.1 mm and a length of few mm.
Purity %	99.999
Crystalline structure	Face centered cubic lattice
Electrical resistivity (Ω cm)	2.06×10^{-6} (25°C)
RRR (300°/4.2°K) (Residual resistance ratio)	1453
Recrystallization temperature	The recrystallization temperature is reported by the manufacturer to be 195°C for a degree of deformation of 70% and 70°C for a degree of deformation of 99.5%. By extrapolation it is assumed that recrystallization takes place at room temperature after a reduction of 99.9%.

An analysis was performed spectrographically with a Hilger Grating Spectrograph at Degussa, which showed the following impurities (ppm):

Fe	0.4 – 2.5
Ag	0.2 – 3.3
Cu	<0.1
Al	0.1 – 0.35
Ni	0.1 – 0.3
Pd	0.1 – 2
Pt	<3
Bi	<2

7.2 Platinum

Manufacturing Process:

Purification of raw platinum was carried out by various chemical procedures and controlled by a *Q* 24 instrument from Zeiss, Jena, with a Feussner spark spectrum. The 99.999% purified platinum then was melted in a ceramic crucible in an argon atmosphere and cast in a water-cooled crucible. After casting about 2 mm of the block were machined off and the block pickled again and drawn in special pure dies being pickled again after about 10 passes. An analysis of a 0.1 mm wire showed an increase in Fe to 10 – 15 ppm and in Au and Cu to about 2 – 3 ppm. Apparently some impurities had entered the material regardless of the pickling.

Since the samples which were required by the different laboratories were of highly varying shape, it was difficult to make a general statement concerning their metallurgical state. All samples in general were provided in a more or less cold-worked state. The cold work amounts only to some percent for samples of several diameters up to more than 90% reduction for samples of 0.5 mm diameter.

Properties:

Density (g/cm³)	21.5
Purity %	99.999
Crystalline structure	Face centered cubic lattice
Electrical resistivity	10×10^{-6} ηcm
RRR (290°/4.2°K)	1300 (wire 0.1 mm dia.)
Recrystallization temperature	250°C (by Vickers hardness measurements)
Tensile strength	31 kg/mm² (5 mm samples) 38 kg/mm² (3 mm samples)

The impurities in the material were found by spectral analysis to be about 1 ppm or lower for Ag, Al, Au, Cu, Fe, Ni, Pd, Rh, Si, Sn, Ti, and Zn.

7.3 Austenitic Alloy

Manufacturing Process:

A 25 kg charge was used to make all samples. To achieve homogeneity the melt was remelted in a vacuum arc-furnace.

After the billets were forged between 1100° and 950°C they were heat-treated for 20 min. at 1050°C and subsequently quenched in water.

For stabilization the material was then submitted to a five hour heat-treatment at 900°C followed by cooling in air.

Properties:

Density (g/cm^3)	8.009
Grain size (ASTM)	3 – 6
Electrical resistivity ($\Omega\, mm^2\, m^{-1}$)	0.81 (100°C) 1.080 (500°C) 1.201 (900°C)
Young's modulus (kp/mm^{-2})	20180 (100°C) 17230 (500°C)
Anisotropy	None
Homogeneity	Variation in content of Ni and Cr of about 0.1 and 0.2%, respectively (tested by means of microprobe analyzer)
Tensile strength (kg/mm^2)	49.6

The material was found to have the following recrystallization behaviour:

Degree of Cold Deformation	Heat Treatment	Microstructure	Grain Size Acc. to ASTM
30%	30 min. 750°C/water quench.	not recrystallization	–
30%	30 min. 800°C/water quench.	beginning recrystallization	8
30%	30 min. 850°C/water quench.	complete recrystallization	6 – 8
60%	30 min. 750°C/water quench.	approximately 60% recryst.	<8
60%	30 min. 800°C/water quench.	nearly compl. recryst.	8
60%	30 min. 850°C/water quench.	complete recrystallization	8
90%	30 min. 750°C/water quench.	almost complete recryst.	<10
90%	30 min. 800°C/water quench.	complete recrystallization	<10
90%	30 min. 850°C/water quench.	complete recrystallization	<8

The recrystallization behaviour was tested on sheet metal samples having an original thickness of 2.3 mm. The test consisted in the following sequence of treatments:

(a) 20 min heat-treatment at 1050°C

(b) Quenching in water

(c) Cold deformation by 30, 60, or 90%.

Of the material thus deformed one sheet metal sample each was heat-treated for 30 min. at the following temperatures:

750, 800, 850°C.

Subsequently each sample was quenched again in water.

In the case of cooling from 1050°C at a cooling rate of approximate 300°C per second the Martensite point lies below minus 196°C.

7.4 Alumina (Al_2O_3)

Manufacturing Process:

For the production of Al_2O_3 material an initial powder consisting of α-Al_2O_3 was used, which was ground to the grain size required for the sintering process. As manufacturing procedure dry-pressing was applied to reach sufficient homogeneity of the samples. Organic binders were used as aid for the pressing process. The samples were continuously controlled for the content of ash (0.04%, of which 50% was MgO).

All samples are pressed at 1000 kg/cm² in matrices consisting of an austenitic alloy. The problem in this process is the possibility of inhomogeneous pressure distribution, especially if the sample lengths are large.

After pressing, the samples were sintered (1800°C/6h) in a furnace, the walls of which were covered by Al_2O_3-material of 99.5%.

Properties:

Density (g/cm³)	3.8 (theoretical: 3.98)
Grain size (um)	10 – 30
Anisotropy	None
Open porosity (%)	0
Rest porosity (%)	5
Crystal structure	α-Al_2O_3 (100%)
Electrical resistivity (η cm)	10^{14} (20°C); 10^6 (1000°C)

The following chemical composition of the material was found (weight %):

Al_2O_3	> 99.5
SiO_2	0.05 – 0.1
MgO	0.2
CaO	0.05
F_2O_3	0.02 – 0.05
Na_2O	0.1 – 0.3

All samples were recrystallized. During annealing for 30 hour at 1650°C in air no grain size variation was observed. At higher temperatures the particle size becomes greater.

According to the supplier the dimensions of the Al_2O_3-samples do not change if the temperature is limited to 1700°C.

7.5 Tungsten

Manufacturing Process:

All tungsten samples were made from the same WO_3-powder batch. To obtain the various sample sizes, the tungsten powder was submitted to a cold compaction in steel molds or to hydrostatic pressing, followed by sintering in dry hydrogen, and by swaging or gorging. According to the various sizes required, the following manufacturing procedures were employed:

The starting powder was compacted under 3.65 tons/cm² to yield bars 16 x 16 x 600 mm. After sintering, the density amounted to 17.8 g/cm² corresponding to a porosity of 7.5%. These bars were then swaged to diameters of 3.3 to 6.8 mm and subsequently ground to final dimensions. The swaging operation reduced the porosity practically to zero.

All samples were distributed without an annealing heat treatment exceeding 1000°C to provide an opportunity to study the effects of recrystallization behaviour.

Properties

Density (g/cm³)	19.2
Purity %	99.98

Analysis of the sample material by the manufacturer yielded the following results (ppm)

Mo	555
Si	54
O_2	20
H_2	2
N_2	4.5
C	10
Fe	25
Zn	0.6
Cu	0.2
Co	0.2
Ni	0.8

7.6 Ta/10 W

Manufacturing Process:

According to the supplier the material was arc-cast and fully annealed.

Properties:

Density (g/cm³)	16.82
Tensile strength at 25°C (psi)	80,000
Thermal conductivity (watt/cm°C)	0.547

7.7 POCO – Graphite AXM 5Q

Manufacturing Process:

Manufacturing methods claimed to be proprietary.

Properties:

The following table gives a survey on properties and characteristics:

Density (g/cm³)	1.70 – 1.78
Anisotropy (unitless) (Property deviation in direction of measurement)	1.03
Purity (ppm)	100 – 200
Particle size (inch)	0.001
Uniformity (dimensionless)	Structure, properties, characteristics are consistent within each piece and from piece to piece.
Porosity (%)	21
Outgassing	Negligible
Process temperature (°C)	2500

The chemical analysis of POCO – graphite AXM 5Q was (ppm):

Fe	100 – 400
Si	80 – 400
Al	20 – 150
V	100 – 400
Ni	10 – 40
Cr	5 – 20
Ti	15 – 20

Traces:

Mg, Cu, B, Mn, Li, Cd, Mo, Pb, Ag, Zn, Ca, P, Si.

7.8 AAQ1 – Graphite

Manufacturing Process:

AAQ1-Graphite was produced from a raw material mix that contained 85 parts by weight of Great Lakes 1008-s-graphite flour, 15 parts by weight of Thermax carbon black, and 27 parts by weight of Varcum 8251 furfuryl alcohol resin. The resin contained 4% by weight of maleic anhydride as a polymerization calatyst. The manufacturing was carried out by using only standard procedures.

For graphitization the baked rods were heated at a nearly constant rate to 2830°C over a period of 6 hr, and furnace-cooled to nearly room temperature in about 3 hr. The result is an extruded, resin-bonded graphite of extreme uniformity.

Properties:

Density (g/cm³)	1.90 ± 0.0028 (average of several lots)
Porosity %	15.9 (calculated from density measurement)
Pore Diameter (μ)	0.4 – 35

Anisotropy - ratio	1.68 (calculated from room temperature measurement of thermal and electrical conductivity)
Purity (ppm)	Ash 300 Fe 30 Si 30 B 1
Electrical resistivity ($\mu\eta$cm)	1150 (average of several lots at room temperature)
Young's Modulus (psi)	2.308×10^6 (average of several lots at room temperature)

7.9 RVD – Graphite

Manufacturing Process:

The RVD-graphite is a petroleum-coke base graphite, fabricated by molding and pressure curing in cylindrical shapes.

The samples used in the present program were taken from a slab of 18 inch diameter by 5 inch thickness.

Properties:

Density (g/cm^3)	1.88 ± 0.013
Anisotropy	Two-fold anisotropic
Grain size (inch)	0.015
Electrical resistivity (Ω cm)	14×15^4 (with grain) 19.6×10^4 (across grain)
Porosity (%)	16 – 17
Process temperature (°C)	2800

7.10 Copper

Manufacturing Process:

The samples were vacuum-cast and exhibit a purity in excess of 99.999%.

The density is 8.96 g/cm^3 as measured by participant No.18.

An spectrographic analysis carried out by the manufacturer yielded the impurities listed below (ppm):

Fe	0.7
Sb	1
Pb	1
Sn	1
Ni	1
Bi	0.1
Ag	0.3
As	2
Cr	0.5
Si	0.1
Te	2

7.11 Silica Glass

It is well known that the use of silica glass at temperatures above 800°C is accompanied by viscous flow and a time-dependent change of thermal expansion. The magnitude of these effects above 800°C will depend on the type of silica glass used and on its special heat treatment for thermal stabilization. If the glass is not stabilized the viscosity at a constant temperature will change with time, always tending to approach its stabilized value. For example (Ref.(8)), an eight-hour heat treatment of the material at 1080°C results in a length change of approximately 0.07 to 0.10% expansion. After such a heat treatment the material does not show any further irreversible thermal expansion behaviour up to temperatures below 1060°C.

Manufacturing Process:

A single rod of 5 mm in diameter was used for the preparation of all samples because of their required conformity. The sample material was heat treated for stabilization, according to ASTM 228-66T specifications. The samples were distributed with rough-cut ends to the participants who finally machined them by polishing.

Properties:

Density (g/cm^3)	2.20
Thermal conductivity (cal/cm °C sec)	0.035 – 0.008 (20° – 800°C)
Specific heat (cal/g °C)	6.5 – 15.5 (20° – 1000°C)
Thermal diffusivity (cm^2/sec)	0.009 – 0.008 (20° – 400°C)
Young's modulus	7350 kp/mm^2

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TABLE 1

Lattice Expansion of Pure Gold, Obtained by Simmons and Balluffi (Ref.(1)), Merryman and Kempter (Ref.(2)) and AGARD Participant No.8, Compared with Bulk Expansion Reported in Ref.(1)

T (°C)	Ref.(1) $\frac{\Delta\alpha}{\alpha_0}$ (%)	Ref.(1) $\frac{\Delta l}{l_0}$ (%)	Ref.(2) $\frac{\Delta\alpha}{\alpha_0}$ (%)	Part. No.8 (AGARD Gold) $\frac{\Delta\alpha}{\alpha_0}$ (%)	Deviation from the mean (%)
20	0	0	0	0	0
100	0.116	0.116	0.110	–	±0.003
200	0.265	0.265	0.255	–	±0.005
300	0.419	0.419	0.416	–	±0.0015
400	0.578	0.578	0.573	–	±0.0025
500	0.745	0.745	0.739	–	±0.0025
600	0.915	0.915	0.912	–	±0.0015
700	1.094	1.095	1.093	–	±0.001
800	1.281	1.285	1.282	1.283	±0.002
900	1.477	1.485	1.484	1.486	±0.0045
1000	1.685	1.701	1.695	1.697	±0.006

TABLE 2

Lattice Expansion of Pure Platinum Obtained by Cambell (Ref.(3)), Compared with Bulk Expansion Reported by Kirby (Ref.(4)) and with Measurements on AGARD Platinum by Participants No.32 and 8

T (°C)	Ref.(3) $\frac{\Delta\alpha}{\alpha_0}$ (%)	Ref.(4) $\frac{\Delta l}{l_0}$ (%)	Part. No.32 AGARD Pt $\frac{\Delta l}{l_0}$ (%)	Part. No.8 AGARD Pt $\frac{\Delta\alpha}{\alpha_0}$ (%)	Deviation from the mean (%)
20	0	0	0	0	0
100	0.072	0.072			0
200	0.165	0.165			0
300	0.260	0.260	It is hoped		0
400	0.357	0.359	to be supplied		±0.001
500	0.457	0.459	later		±0.001
600	0.560	0.565			±0.0025
700	0.665	0.670			±0.0025
800	0.774	0.777	0.783	0.770	±0.0065
900	0.885	0.891	0.894	0.887	±0.0065
1000	0.999	1.005	1.012	0.995	±0.0085

TABLE 3

Survey of Pushrod Dilatometer Characteristics

Partic. No.	Reference material system of the pushrod dilatometer	Measurement of the length change	Registration of measuring data
2	quartz glass	m.o.	photograph. film
5	Al_2O_3	e.i.	recorder
6	quartz glass	m.o.	photograph. film
7	quartz glass	m.o.	photograph. film
8	quartz glass	e.i.	recorder
9	Al_2O_3	e.i.	recorder
12	quartz glass	e.i.	recorder
14	Al_2O_3	e.i.	recorder
18	Al_2O_3	e.i.	recorder
21	quartz glass	e.i.	recorder
29	quartz glass	e.i.	recorder
30	quartz glass	e.i.	recorder
45	quartz glass	m.o.	photograph. film

e.i. = electromagnetic induction
m.o. = mechanical/optical

TABLE 4

Experimental Results of the Thermal Expansion as Obtained on Gold

Partic. No. / T°C	2		5		6		7		8		9		14		29		30		45		
	a	b	a	b	a	b	a	b	a	b	a	b	a	b	a	b	a	b	a	b	c
20	0	–	0	–	0	–	0	–	0	–	0	–	0	–	0	–	0	–	0	–	
100	–		0.105		–		0.135		0.100		0.097		0.112		0.112		0.115		0.122		
200	–		0.250		–		0.280		0.240		0.236		0.256		0.257		0.291		0.260		
300	0.421	±0.006	0.415		0.424	±0.003	0.430		0.410	±0.005	0.412	±0.001	0.407	±0.007	0.415		0.426		0.416		0.418
400	0.579		0.580		0.582		0.580		0.560		0.575		0.566		0.574		0.605		0.571		0.577
500	0.746		0.750		0.746		0.740		0.750		0.742		0.729		0.734		0.774		0.737	±0.010	0.745
600	0.919	±0.007	0.930		0.917	±0.003	0.905		0.930	±0.006	0.931	±0.004	0.903	±0.007	0.906		0.944		0.909		0.919
700	1.097		1.110		1.090		1.080		1.120		1.110		1.085		1.080		1.110		1.089		1.097
800	1.281		1.280		1.275		1.265		1.310		1.295		1.277		1.275		1.304		1.276		1.265
900	1.481	±0.008	1.480		1.471	±0.006	1.440		1.510	±0.008	1.500	±0.007	1.480	±0.009	1.470		–		1.473		1.479
1000	–		–		–		–		–		–		–		–		–		1.675		–

a = individual average value $\Delta l/l_0$ (%)

b = absolute maximum deviation from the the individual mean, $\Delta(\Delta l/l_0)$ (%)

c = group mean, $\Delta l/l_0$ (%)

TABLE 5

Experimental Results of the Thermal Expansion as Obtained on Platinum

Partic. No. / T°C	2		5		6		8		9		12		14		21		30		32		
	a	b	a	b	a	b	a	b	a	b	a	b	a	b	a	b	a	b	a	b	c
20	0		0		0		0		0		0		0		0		0		0		–
100	–		0.070		–		–		0.060		0.067		0.072		0.076		0.075				–
200	–		0.165		–		–		0.145		0.164		0.162		0.166		0.168				–
300	0.261	±0.006	0.270		0.264	±0.002	0.254	±0.007	0.260		0.266		0.254	±0.007	0.259	±0.006	0.267				0.262
400	0.362		0.375		0.362		0.361		0.368	±0.003	0.358		0.352		0.356		0.371				0.363
500	0.466		0.480		0.461		0.466		0.469		0.455	<±0.01	0.454		0.452		0.474				0.464
600	0.569	±0.004	0.590		0.563	±0.002	0.575	±0.008	0.575		0.559		0.561	±0.007	0.553	±0.008	0.577				0.596
700	0.672		0.700		0.668		0.689		0.680	±0.003	0.672		0.672		0.662		0.684				0.678
800	0.780		0.810		0.772		0.806		0.780		0.771		0.786		0.780		0.793		0.783		0.786
900	0.903	±0.005	0.920		0.888	±0.0025	0.926	±0.008	0.892		0.884		0.901	±0.008	0.900		0.898		0.894		0.901
1000	1.013		1.030		1.015		–		0.993	±0.003	0.993		1.017		1.021	±0.013	–		1.012		1.010

a = individual average value $\Delta l/l_0$ (%)
b = absolute maximum deviation from the individual mean, $\Delta(\Delta l/l_0)$ (%)
c = group mean $\Delta l/l_0$ (%)

TABLE 6

Experimental Results on Thermal Expansion as Obtained on the Austenitic Alloy

Partic. No. / T°C	5		6		7		8		9		21		29		30		
	a	b	a	b	a	b	a	b	a	b	a	b	a	b	a	b	c
20	0		0		0		0		0		0		0		0		–
100	0.120		–		0.138		0.098		0.110		–		–		0.126		–
200	0.300		–		0.318		0.262		0.280		–		0.300		0.294		–
300	0.480		0.497	±0.00	0.507		0.483	±0.006	0.490	±0.004	–		0.450		0.500		0.482
400	0.680		0.687		0.700		0.688		0.680		0.685		0.682		0.697		0.657
500	0.880		0.883	±0.002	0.890		0.894		0.899		0.877		0.883		0.888		0.887
600	1.100		1.082		1.100		1.086	±0.007	1.070	±0.006	1.076		1.100		1.100		1.089
700	1.320		1.285		1.300		1.316		1.280		1.282		1.315		1.310		1.301
800	1.520		1.494		1.510		1.515		1.490		1.490		1.530		1.530		1.510
900	1.720		1.710		1.730		1.750	±0.010	1.710	±0.004	1.710		1.740		1.750		1.727
1000	1.920		1.936	±0.00	–		1.974		1.910		1.924		1.960		1.960		1.941

a = individual average value $\Delta l/l_0$ (%)
b = absolute maximum deviation from the individual mean, $\Delta(\Delta l/l_0)$ (%)
c = group mean, $\Delta l/l_0$ (%)

TABLE 7

Experimental Results of the Thermal Expansion as Obtained on Alumina

Partic. No. / T°C	5		6		8		9		12		14		18		29		30		
	a	b	a	b	a	b	a	b	a	b	a	b	a	b	a	b	a	b	c
20	0		0		0		0		0		0		0		0		0		–
100	0.030		–		0.039		0.039		0.038		0.045		0.047		–		0.0474		–
200	0.105		–		0.105		0.093		0.105		0.112		0.112		0.110		0.118		–
300	0.200		0.189	±0.0005	0.186	±0.007	0.188	±0.001	0.184		0.184	±0.006	0.189	±0.004	0.190		0.197		0.190
400	0.280		0.265		0.270		0.271		0.271		0.264		0.263		0.270		0.278		0.270
500	0.380		0.348		0.348		0.356		0.348	<±0.006	0.349		0.347		0.350		0.364		0.354
600	0.460		0.434	±0.0005	0.450	±0.008	0.446	±0.005	0.440		0.440		0.434	±0.005	0.440		0.452		0.444
700	0.550		0.522		0.544		0.535		0.535		0.532		0.524		0.530		0.541		0.535
800	0.640		0.614		0.645		0.620		0.623		0.628		0.609		0.620		0.634		0.626
900	0.720		0.709	±0.001	0.757	±0.009	0.715	±0.004	0.712		0.724		0.709	±0.005	0.715		0.729		0.721
1000	0.810		0.808		0.854		0.800		0.800		0.820	±0.007	0.806		0.790		0.815		0.812

a = individual average value $\Delta l/l_0$ (%)
b = absolute maximum deviation from the individual mean, $\Delta(\Delta l/l_0)$ (%)
c = group mean, $\Delta l/l_0$ (%)

TABLE 8

Experimental Results of the Thermal Expansion as Obtained on Ta/10 W

Partic. No. / T°C	2		6		29		30		
	a	b	a	b	a	b	a	b	c
20	0		0		0		0		–
100	–		–		0.034		0.0435		–
200	–		–		0.085		0.109		–
300	0.188	±0.005	0.187	±0.00	0.144		0.172		0.173
400	0.257		0.247		0.203		0.239		0.237
500	0.325		0.313		0.266		0.308		0.303
600	0.388	±0.005	0.379	±0.00	0.333		0.380		0.370
700	0.455		0.448		0.407		0.456		0.442
800	0.530		0.522		0.430		0.529		0.503
900	0.604	±0.006	0.595	±0.00	0.555		0.605		0.590
1000	0.685		0.674		–		0.683		0.681

a = individual average value $\Delta l/l_0$ (%)
b = absolute maximum deviation from the individual mean, $\Delta(\Delta l/l_0)$ (%)
c = group mean, $\Delta l/l_0$ (%)

TABLE 9

Experimental Results of the Thermal Expansion as Obtained on Tungsten

Partic. No. / T°C	5		6		8		14		18		21		30		
	a	b	a	b	a	b	a	b	a	b	a	b	a	b	c
20	0		0		0		0		0		0		0		–
100	0.030		–		0.034		0.035		0.040		–		–		–
200	0.075		–		0.094		0.078		0.090		–		–		–
300	0.120		0.129	±0.000	0.149	±0.011	0.122	±0.006	0.142	±0.004	0.115	±0.015	0.129		0.129
400	0.175		0.177		0.203		0.169		0.190		0.158		0.181		0.178
500	0.230		0.226		0.262		0.218		0.247		0.204		0.232		0.231
600	0.280		0.275	±0.00	0.319	±0.008	0.270	±0.006	0.304	±0.005	0.249	±0.015	0.287		0.283
700	0.330		0.325		0.382		0.324		0.363		0.298		0.339		0.337
800	0.380		0.377		0.447		0.380		0.420		0.347		0.390		0.393
900	0.425		0.429	±0.005	0.515	±0.004	0.434	±0.006	0.480	±0.005	0.395	±0.014	0.442		0.446
1000	0.480		0.483		0.587		0.486		0.540		0.447		–		0.504

a = individual average value $\Delta l/l_0$ (%)
b = absolute maximum deviation from the individual mean, $\Delta(\Delta l/l_0)$ (%)
c = group mean, $\Delta l/l_0$ (%)

TABLE 10

Experimental Results of the Thermal Expansion as Obtained on AAQ1-Graphite

Partic. No. / T°C	6		8		29		30		32		45		
	a	b	a	b	a	b	a	b	a	b	a	b	c
20	0		0		0		0		0		0		–
100	–		–		–		0.016		–		0.012		–
200	–		–		0.042		0.039		–		0.030		–
300	0.054	±0.001	0.046	±0.006	0.070		0.065		–		0.053		0.057
400	0.078		0.073		0.100		0.094		–		0.080		0.085
500	0.105		0.101		0.132		0.127		–		0.108	±0.007	0.115
600	0.135	±0.001	0.133	±0.010	0.152		0.160		–		0.138		0.144
700	0.167		0.168		0.195		0.195		0.161		0.170		0.176
800	0.202		0.209		0.230		0.230		0.195		0.206		0.212
900	0.237	±0.002	0.250	±0.010	0.250		0.266		0.231		0.243		0.246
1000	0.276		0.296		0.280		0.302		0.266		0.282		0.284

a = individual average value $\Delta l/l_0$ (%)
b = absolute maximum deviation from the individual mean, $\Delta(\Delta l/l_0)$ (%)
c = group mean, $\Delta l/l_0$ (%)

TABLE 11

Experimental Results of the Thermal Expansion as Obtained on POCO Graphite

Partic. No. / T°C	6		8		16		29		32		45		46		
	a	b	a	b	a	b	a	b	a	b	a	b	a	b	c
20	0	–	0	–	0	–	0	–	0	–	0	–	0	–	–
100	–		–		0.080		0.050		–		0.057		0.056		
200	–		–		0.160		0.120		–		0.128		0.127		
300	0.203	±0.00	0.191	±0.004	0.200		0.190		–		0.203		0.202		0.198
400	0.277		0.271		0.280		0.270		–		0.282		0.279		0.277
500	0.355		0.356		0.360		0.355		–		0.362	<±0.006	0.358		0.358
600	0.436	±0.00	0.445	±0.007	0.445		0.445		–		0.447		0.440		0.443
700	0.519		0.540		0.530		0.525		0.467		0.534		0.524		0.520
800	0.607		0.635		0.600		0.600		0.544		0.623		0.610		0.603
900	0.697	±0.00	0.737	±0.011	0.710		0.685		0.620		0.717		0.698		0.695
1000	0.793		0.836		0.770		–		0.705		0.818		0.788		0.792

a = individual average value $\Delta l/l_0$ (%)
b = absolute maximum deviation from the individual mean, $\Delta(\Delta l/l_0)$ (%)
c = group mean, $\Delta l/l_0$ (%)

TABLE 12

Experimental Results of the Thermal Expansion as Obtained on RVD Graphite (with grain)

Partic. No. / T°C	6		8		16		29		45		
	a	b	a	b	a	b	a	b	a	b	c
20	0		0		0		0		0		–
100	–		–		0.040		0.022		0.023		–
200	–		–		0.060		0.056		0.058		–
300	0.107	±0.005	0.098	±0.006	0.100		0.094		0.096		0.099
400	0.146		0.142		0.150		0.135		0.137		0.142
500	0.189		0.189		0.200		0.178		0.181	<±0.007	0.187
600	0.234	±0.005	0.238	±0.005	0.230		0.222		0.229		0.231
700	0.280		0.289		0.280		0.267		0.276		0.278
800	0.329		0.343		0.330		0.300		0.327		0.326
900	0.380	±0.005	0.396	±0.007	0.370		0.356		0.382		0.377
1000	0.433		0.468		0.420		–		0.439		0.440

a = individual average value $\Delta l/l_0$ (%)
b = absolute maximum deviation from the individual mean, $\Delta(\Delta l/l_0)$ (%)
c = group mean, $\Delta l/l_0$ (%)

TABLE 13

Experimental Results of the Thermal Expansion as Obtained on RVD Graphite (across grain)

Partic. No. / T°C	6		8		16		29		45		
	a	b	a	b	a	b	a	b	a	b	c
20	0		0		0		0		0		–
100	–		–		–		0.018		0.023		–
200	–		–		0.07		0.049		0.054		–
300	0.096	±0.005	0.088	±0.008	0.110		0.082		0.094		0.094
400	0.133		0.129		0.150		0.119		0.129		0.132
500	0.173		0.174		0.195		0.156		0.171	<±0.007	0.174
600	0.215	±0.005	0.222	±0.006	0.220		0.195		0.216		0.215
700	0.259		0.270		0.264		0.235		0.262		0.259
800	0.305		0.321		0.318		0.277		0.313		0.307
900	0.352	±0.005	0.375	±0.005	0.38		0.321		0.366		0.359
1000	0.403		0.430		0.43		–		0.420		0.421

a = individual average value $\Delta l/l_0$ (%)
b = absolute maximum deviation from the individual mean, $\Delta(\Delta l/l_0)$ (%)
c = group mean, $\Delta l/l_0$ (%)

TABLE 14

Experimental Results of the Thermal Expansion as Obtained on Copper

Partic. No. / T°C	5		6		8		9		18		21		29		
	a	b	a	b	a	b	a	b	a	b	a	b	a	b	c
20	0	–	0	–	0	–	0	–	0	–	0	–	0	–	–
100	0.130		–		–		0.128		0.135		–				–
200	0.300		–		0.250		0.304		0.302		0.305				–
300	0.480		0.508	±0.008	0.417	±0.014	0.493	±0.002	0.460		0.488		0.490		0.477
400	0.630		0.694		0.618		0.688		0.628		0.675		0.670		0.657
500	0.830		0.851		0.832		0.893		0.810		0.876		0.880		0.859
600	1.070		1.095	±0.007	1.036	±0.032	1.095	±0.014	0.975		1.080		1.110		1.066
700	1.280		1.309		1.271		1.287		1.160		1.295		1.340		1.279
800	1.500		1.535		1.510		1.518		1.380		1.522		1.590		1.508
900	1.740		1.771	±0.015	1.760	±0.024	1.741	±0.002	1.595		1.762		1.840		1.746
1000	–		–		2.014		1.972		1.820		–		–		1.935

a = individual average value $\Delta l/l_0$ (%)
b = absolute maximum deviation from the individual mean, $\Delta(\Delta l/l_0)$ (%)
c = group mean, $\Delta l/l_0$ (%)

TABLE 15

Experimental Results of the Thermal Expansion as Obtained on Quartz

Partic. No. / T°C	6		8		9		12		14		18		21		29		30		42		
	a	b	a	b	a	b	a	b	a	b	a	b	a	b	a	b	a	b	a	b	c
20	0	–	0		0	–	0	–	0	–	0	–	0	–	0	–	0	–	0	–	–
100	–		–		0.0041		0.0040		–		–		–		–		0.0031		0.0025		–
200	0.0097		0.0097		0.0100		0.0070		–		–		–		–		0.0092		0.0053		–
300	0.0153		0.0153	±0.004	0.0162	±0.005	0.0116		0.0124	±0.005	0.0100		0.0330		0.0150		0.0134		0.0100		0.0152
400	0.0209		0.0209		0.0225		0.0152		0.0185		0.0180		0.0410		0.0210		0.0185		0.0138		0.0210
500	0.0266		0.0266		0.0286		0.0186		0.0260		0.0310		0.0490		0.0260		0.0238		0.0174		0.0273
600	0.0324		0.0328	±0.004	0.0346	±0.007	0.0202	<±0.009	0.0357	±0.005	0.0440		0.0560		0.0300		0.0290		0.0211		0.0336
700	0.0377		0.0378		0.0404		0.0210		0.0456		0.0600		0.0620		0.0330		0.0341		0.0297		0.0401
800	0.0428		0.0428		0.0460		0.0216		0.0567		0.0700		0.0680		0.0360		0.0392		0.0283		0.0451
900	0.0480		0.0478	±0.005	0.0515	±0.008	0.0234		0.0655	±0.005	–		0.0750		0.0390		0.0440		0.0318		0.0473

a = individual average value $\Delta l/l_0$ (%)
b = absolute maximum deviation from the individual mean, $\Delta(\Delta l/l_0)$ (%)
c = group mean, $\Delta l/l_0$ (%)

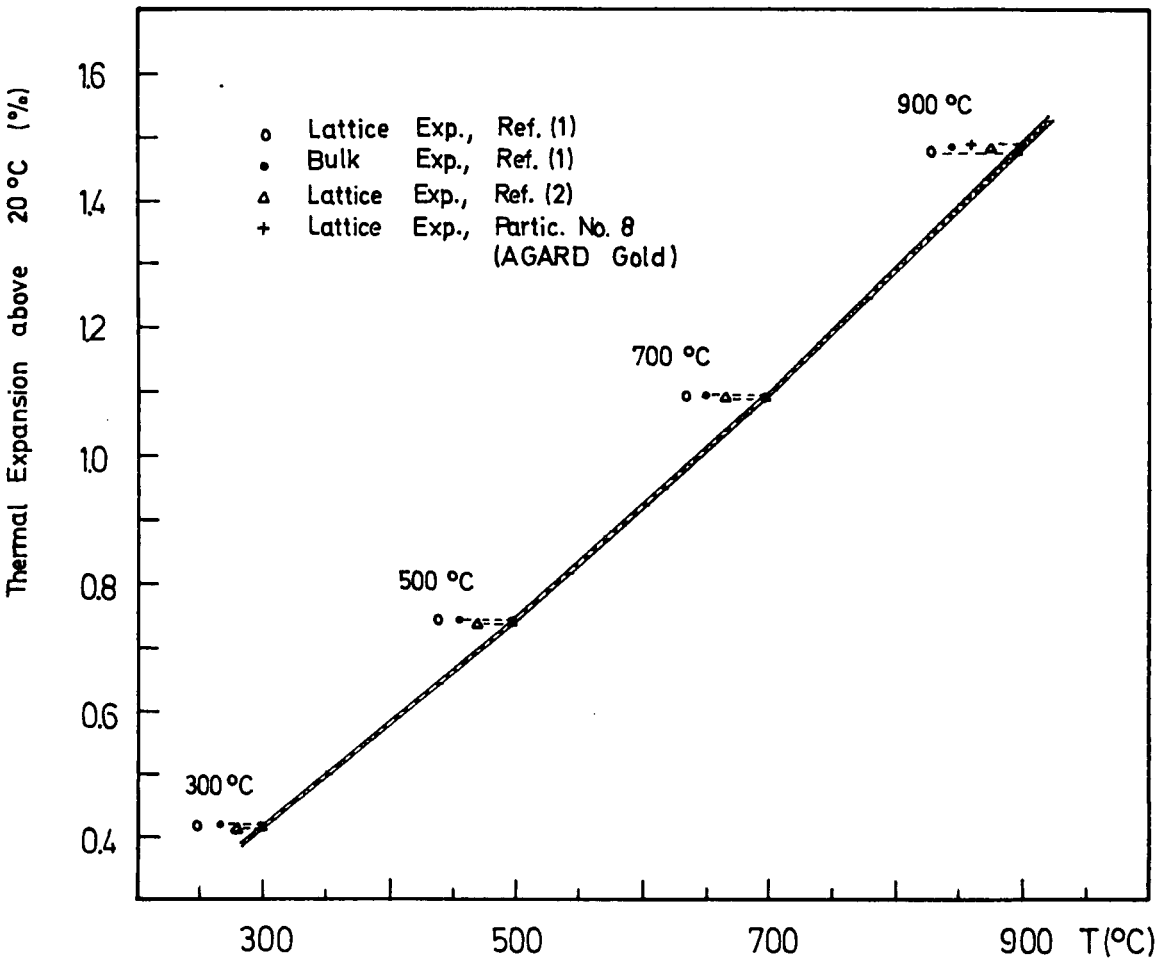

Fig.1 Thermal expansion of pure gold (compare Table 1)

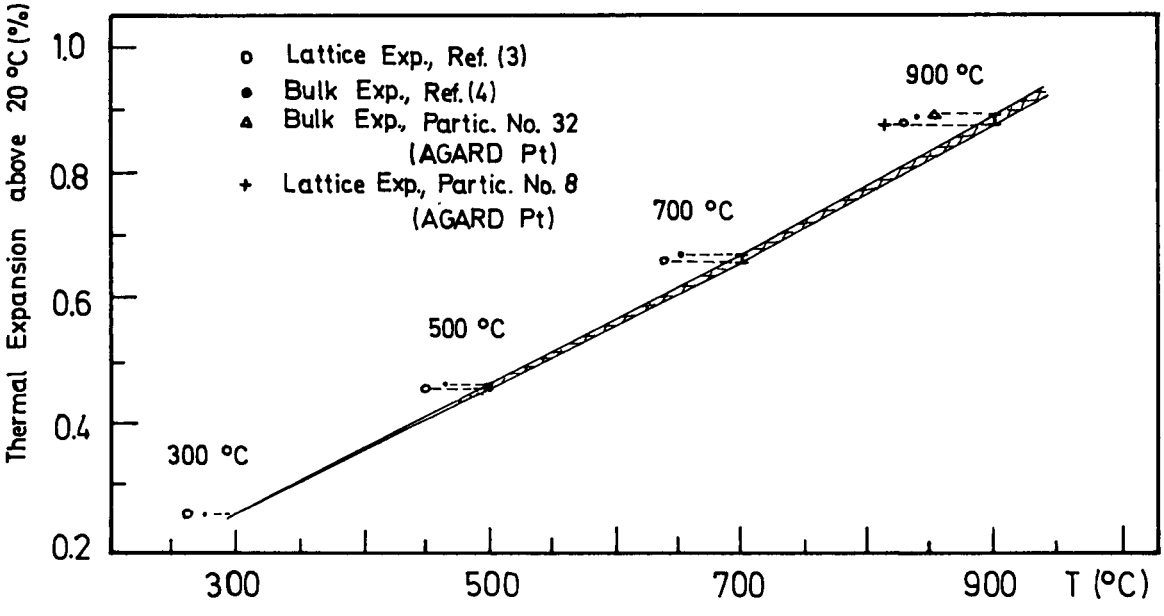

Fig.2 Thermal expansion of pure platinum (compare Table 2)

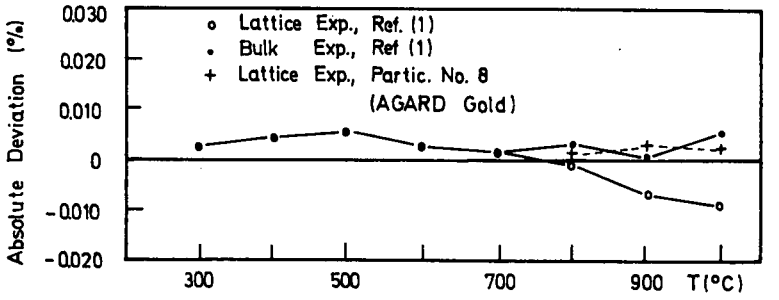

Fig.3 Deviations of the values on thermal expansion of gold (Table 1) using the data due to Merryman and Kempter (Ref.(2)) as the zero line

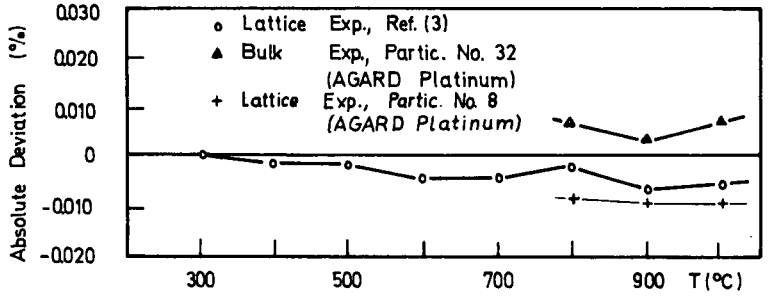

Fig.4 Deviations of the values on thermal expansion of platinum (Table 2) using the data due to Kirby (Ref.(4)) as the zero line

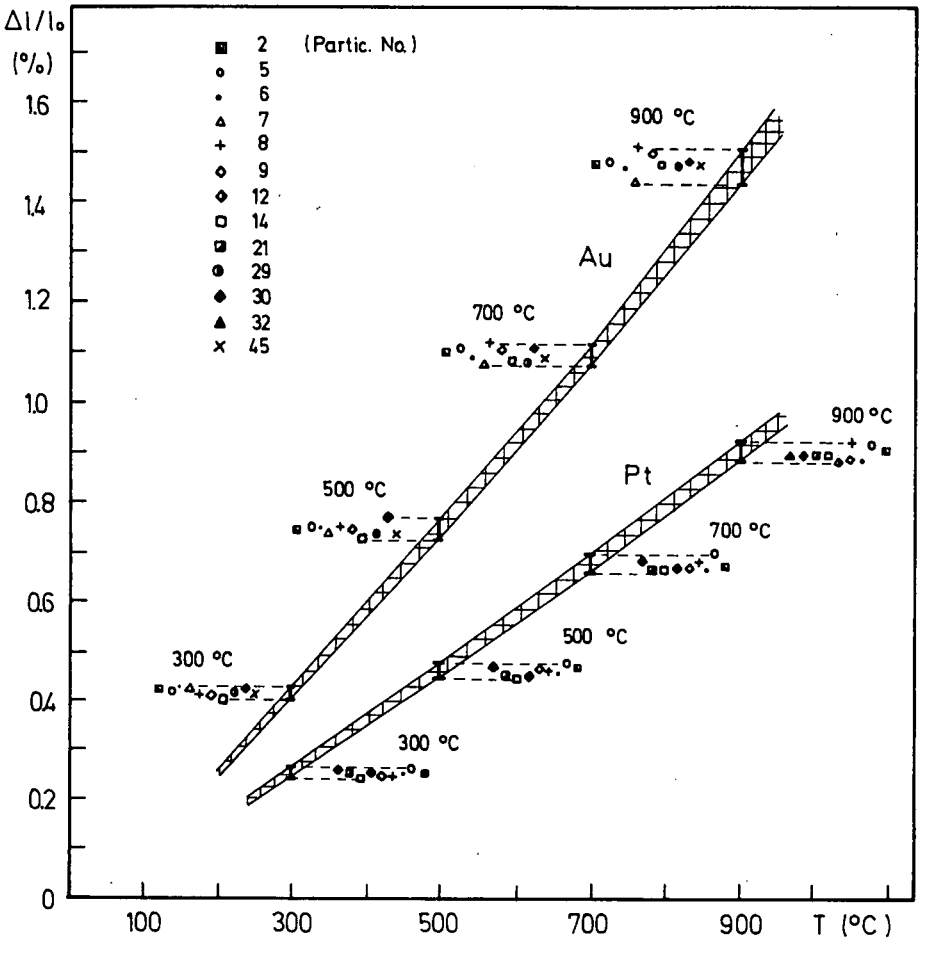

Fig.5 Experimental results of AGARD gold and AGARD platinum as obtained by various participants

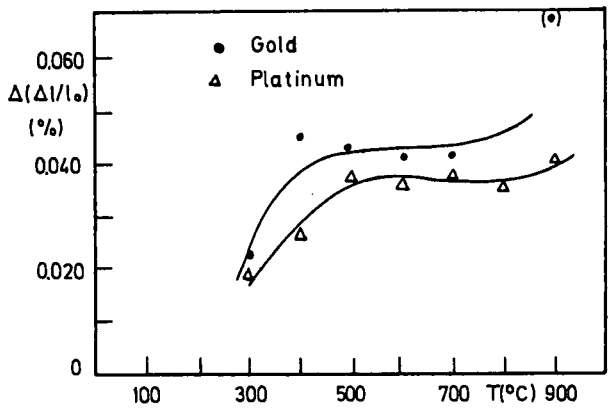

Fig.6 Group scatter $\Delta(\Delta l/l_0)$ as a function of temperature for gold and platinum

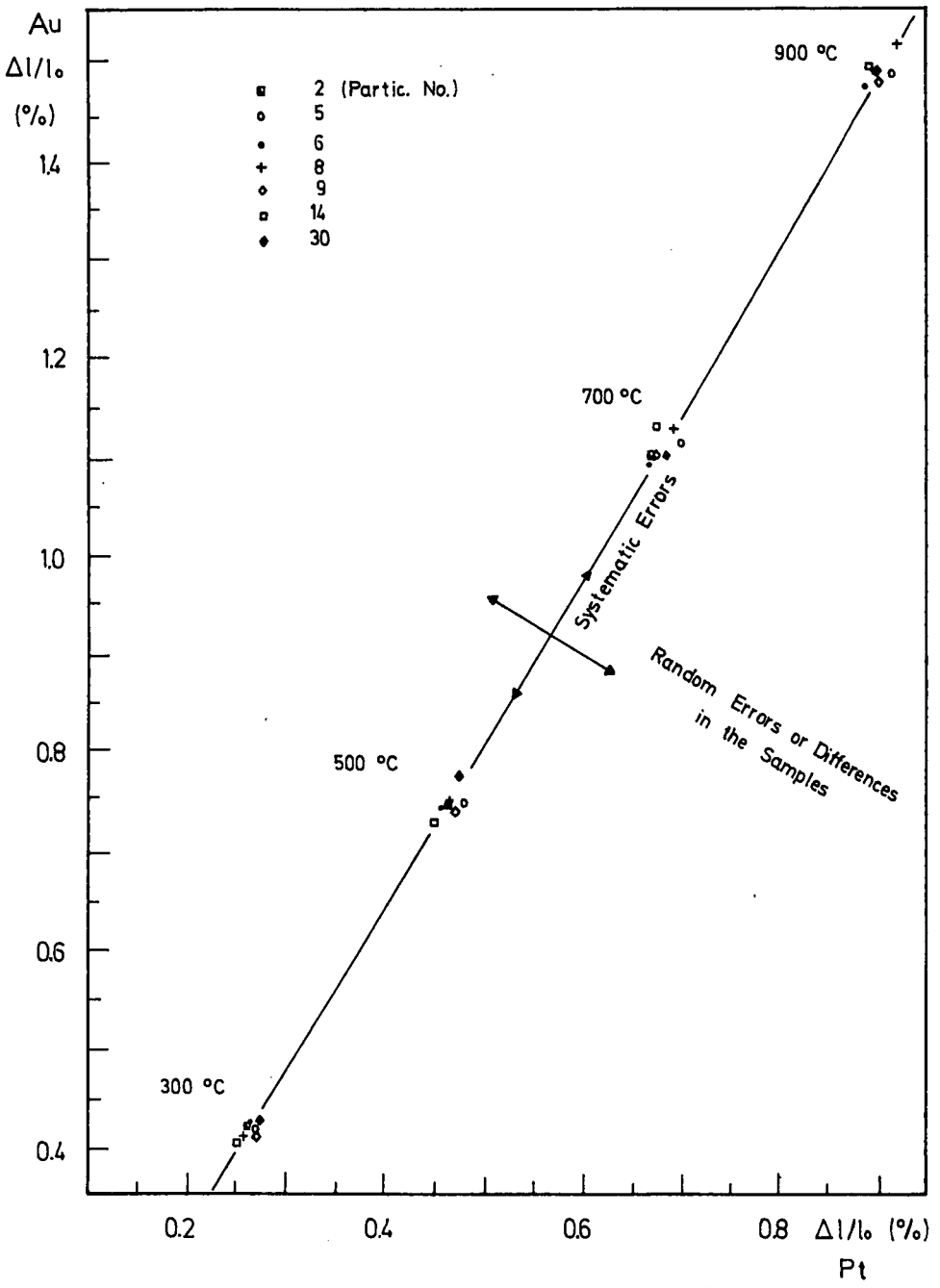

Fig.7 Error Analysis plot based on data for gold and platinum

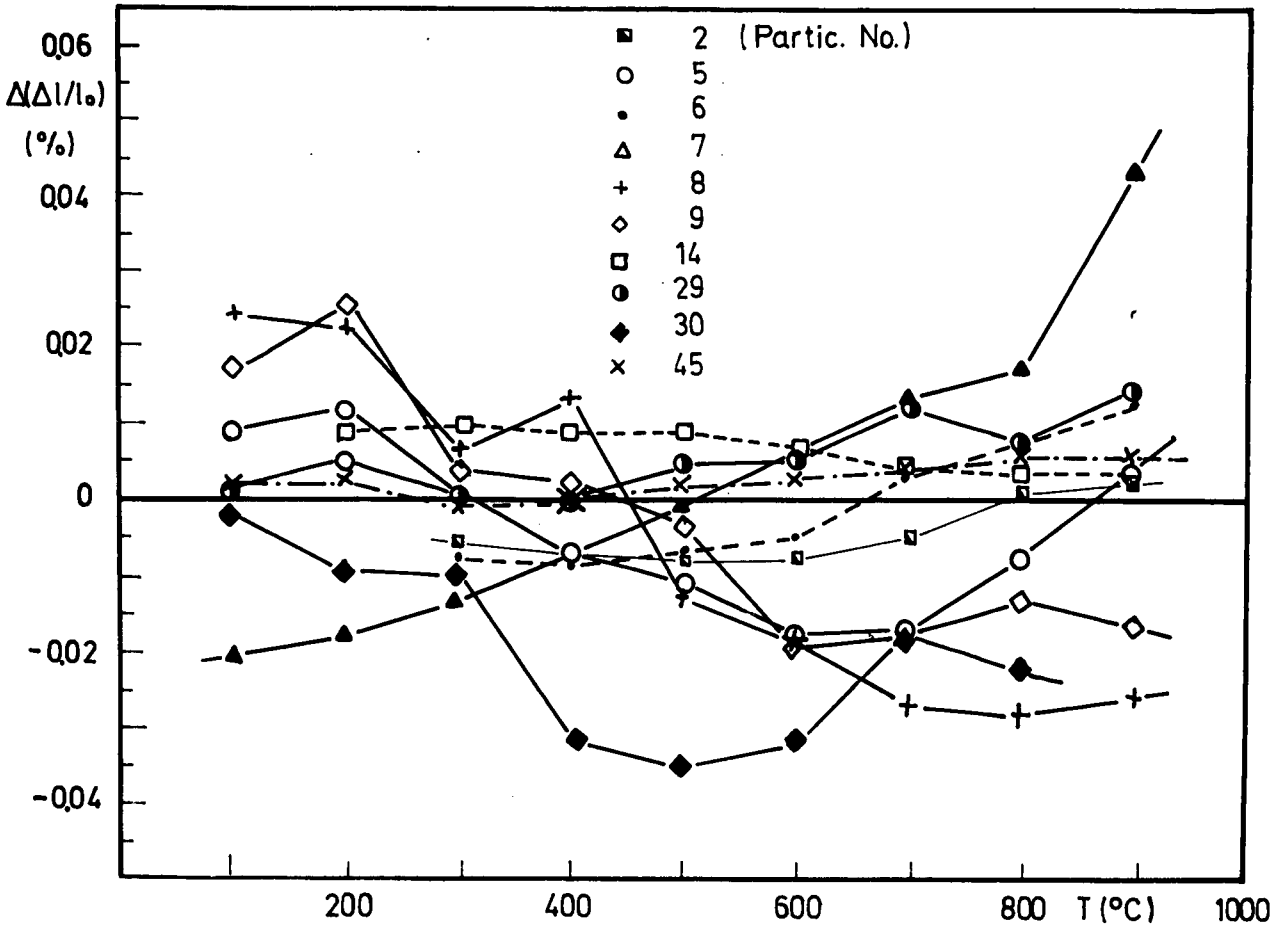

Fig.8 Correction terms $\Delta(\Delta l/l_0)$ for various dilatometers as obtained on gold

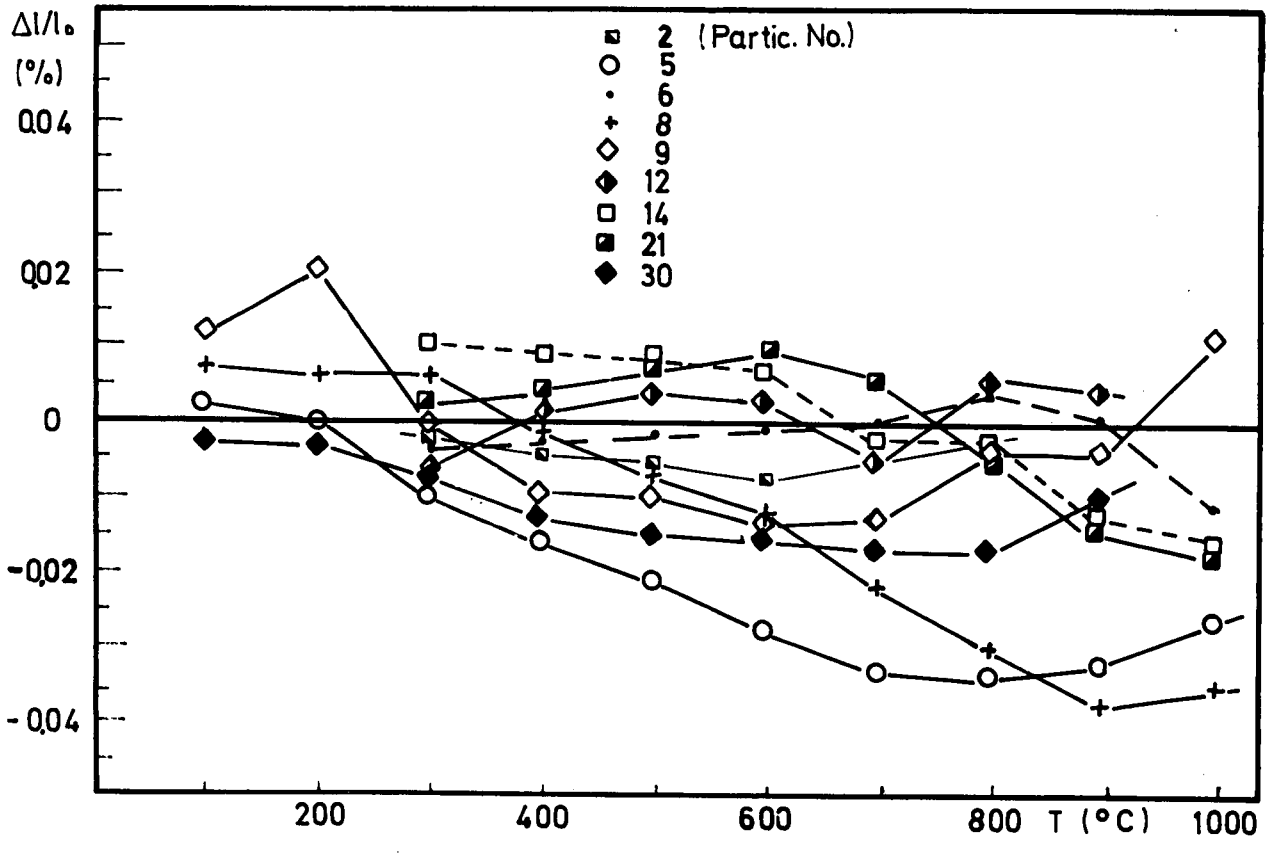

Fig.9 Correction terms $\Delta(\Delta l/l_0)$ for various dilatometers as obtained on platinum

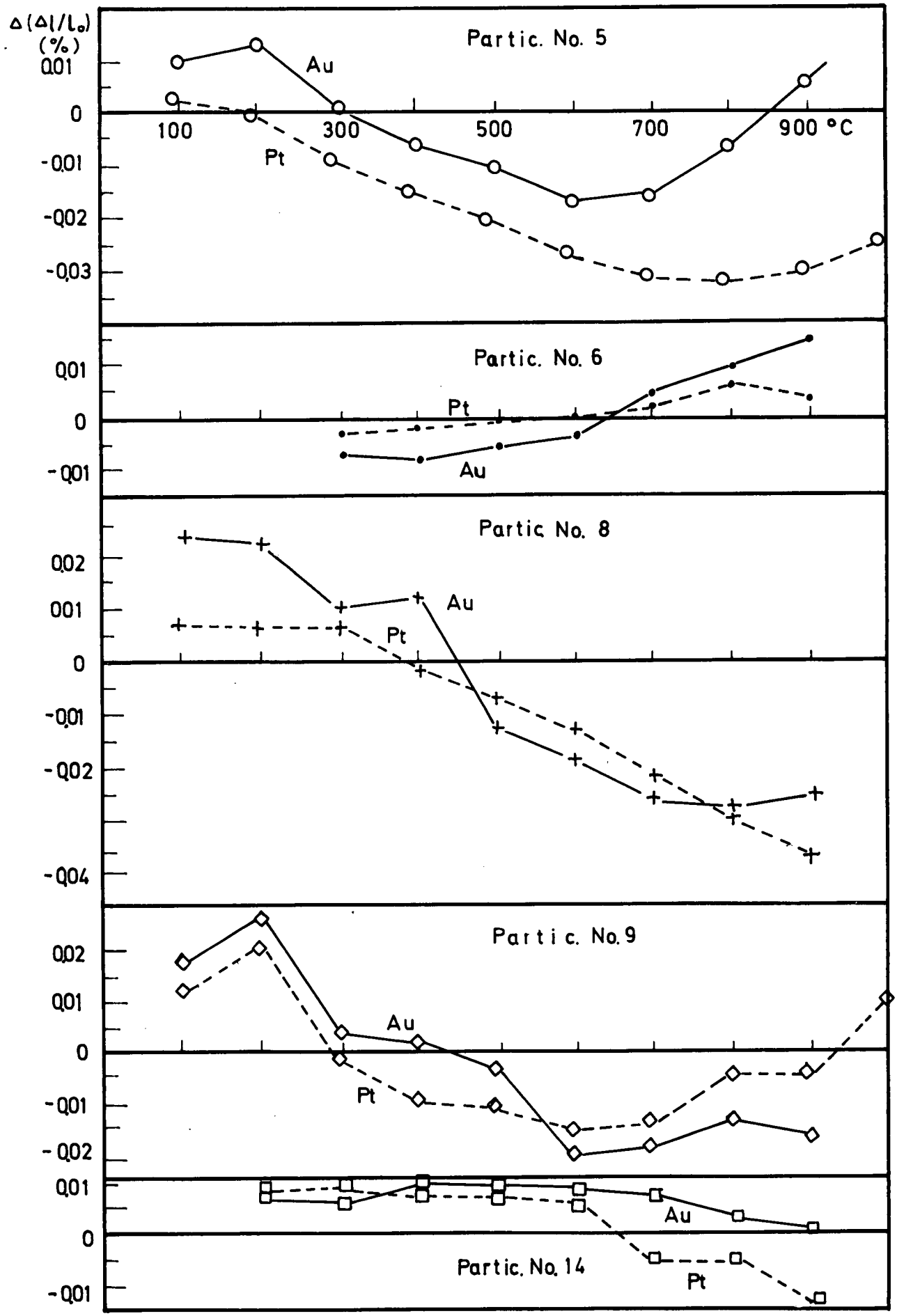

Fig. 10 Comparison of the correction terms $\Delta(\Delta l/l_0)$ as obtained on gold and platinum

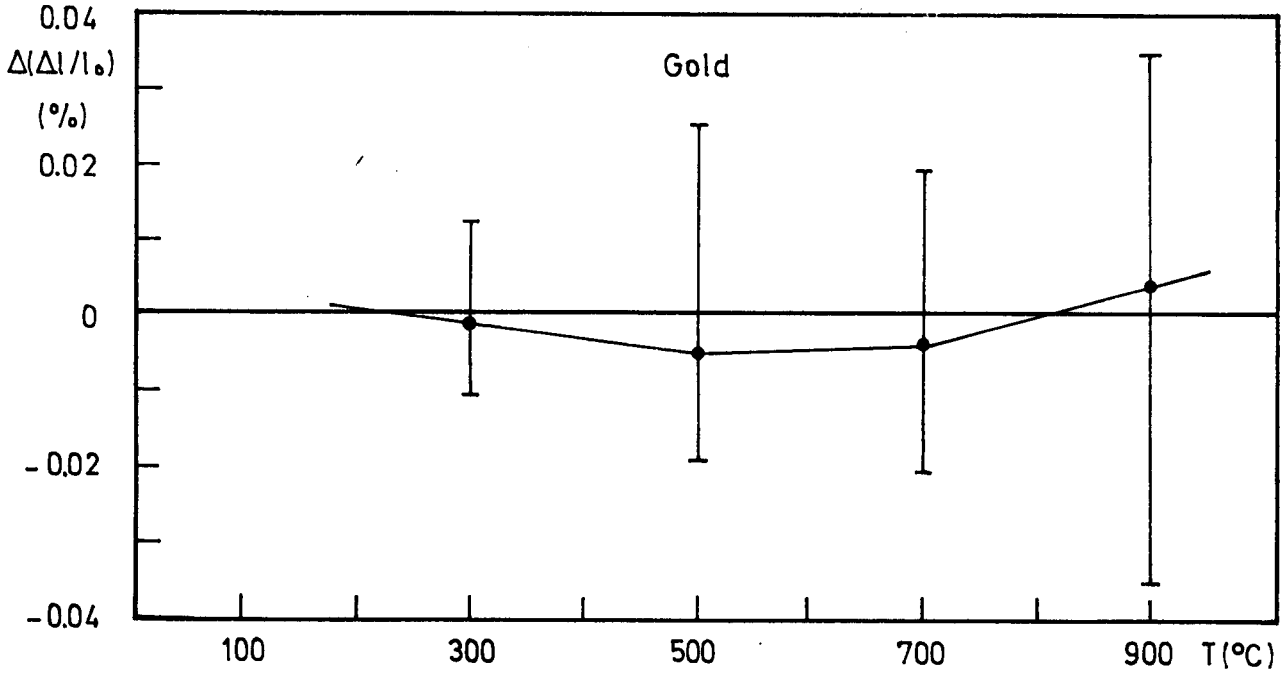

Fig.11 Deviation of the arithmetic mean of all experimental results on gold from the absolute values as listed in Table 1, Ref.(2). The band width represents the group scatter relative to the arithmetic mean

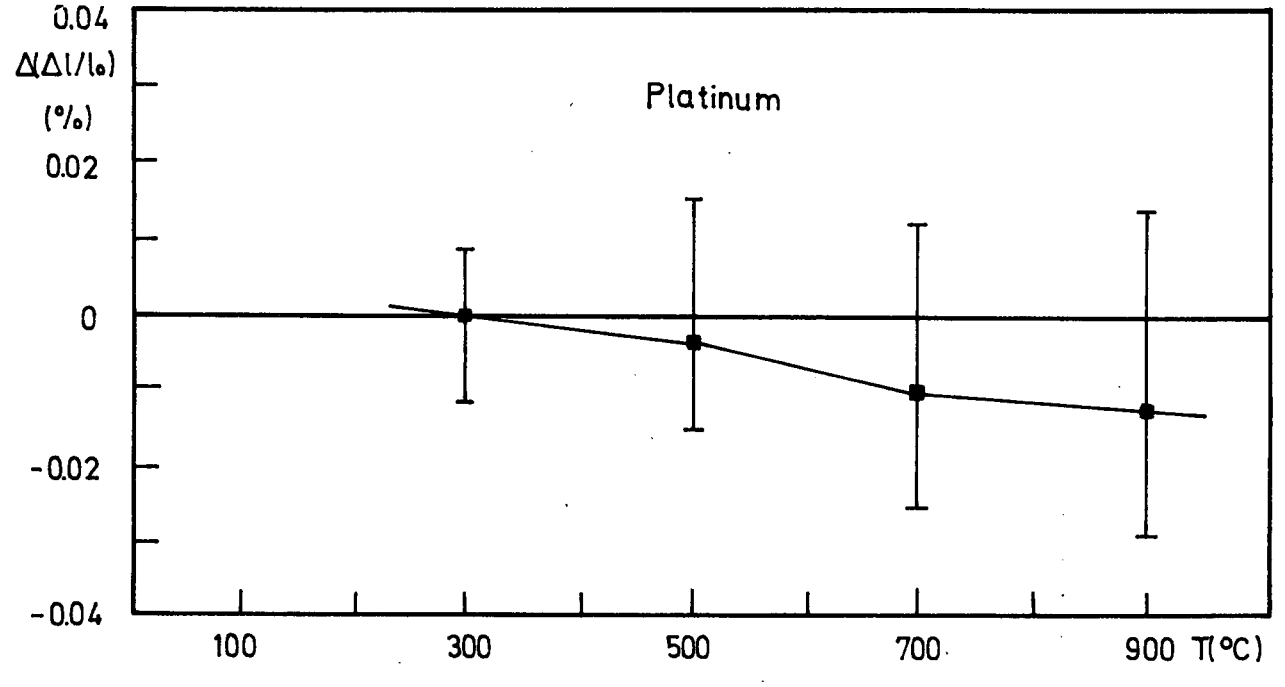

Fig.12 Deviation of the arithmetic mean of all experimental results on platinum from the absolute values as listed in Table 2, Ref.(4). The band width represents the group scatter relative to the arithmetic mean

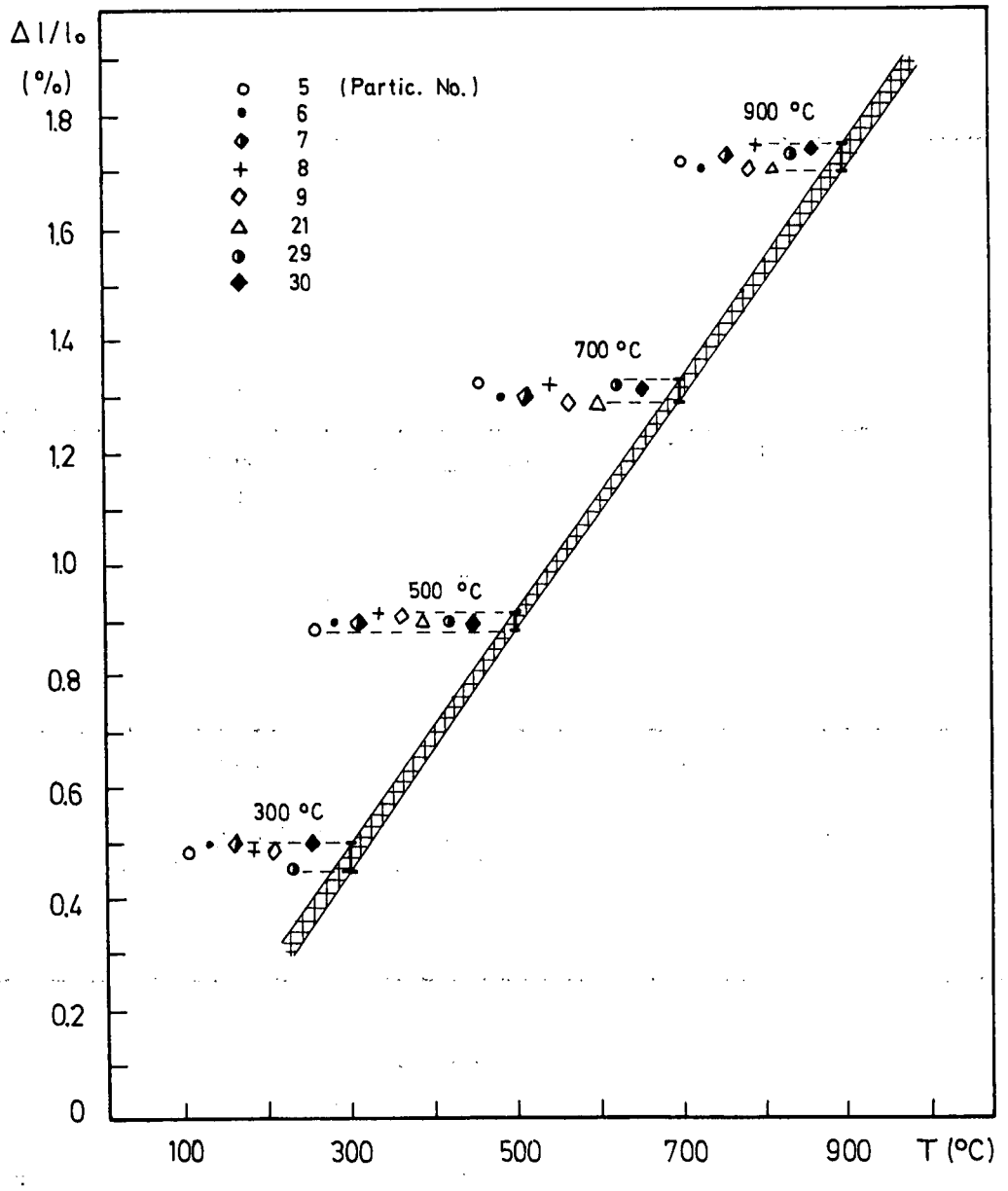

Fig.13 Group scatter of the austenitic alloy

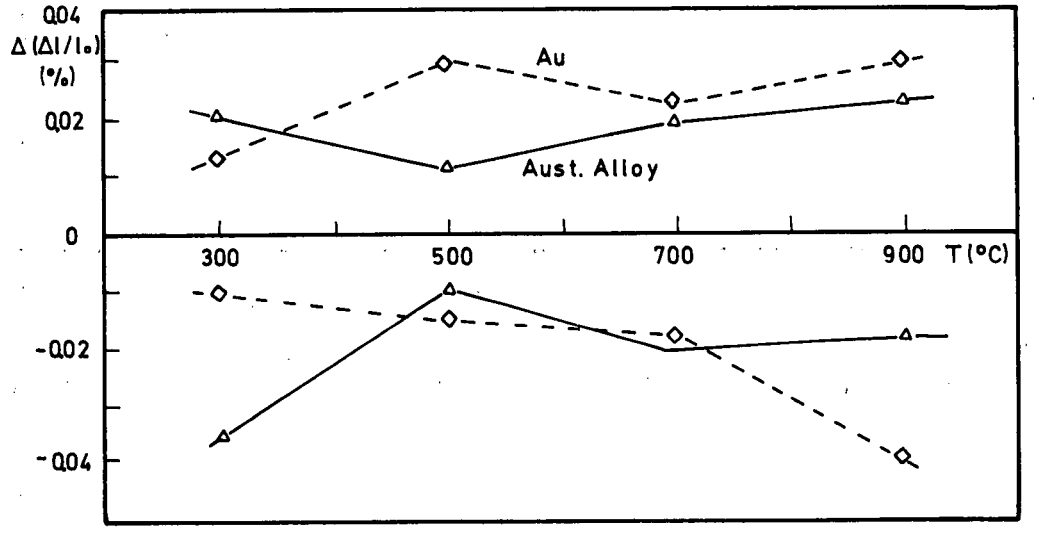

Fig.14 Group scatter obtained for the austenitic alloy compared with the group scatter obtained for gold. The zero line represents the group mean

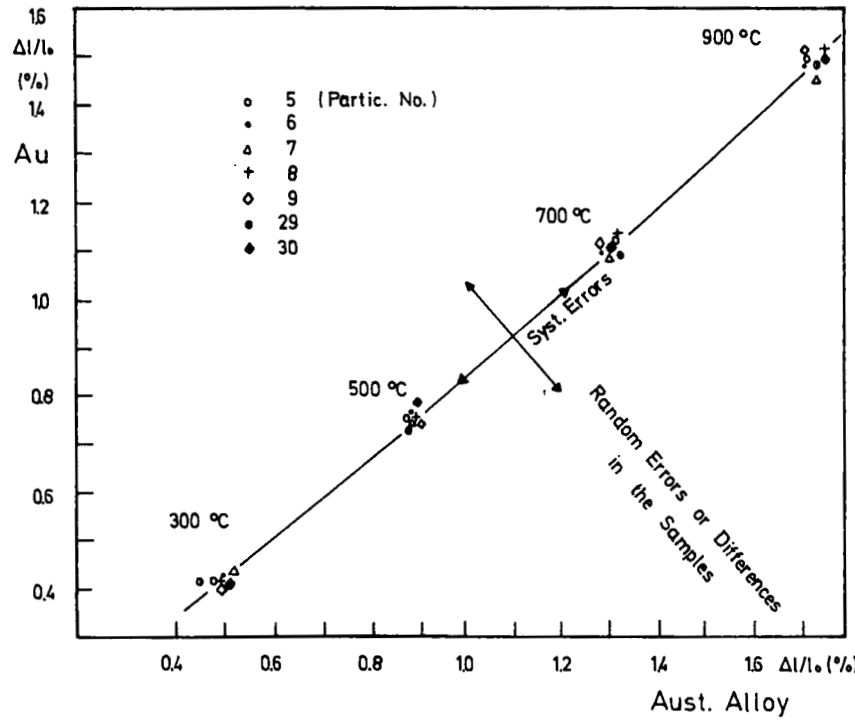

Fig. 15 Error analysis plot austenitic alloy versus gold

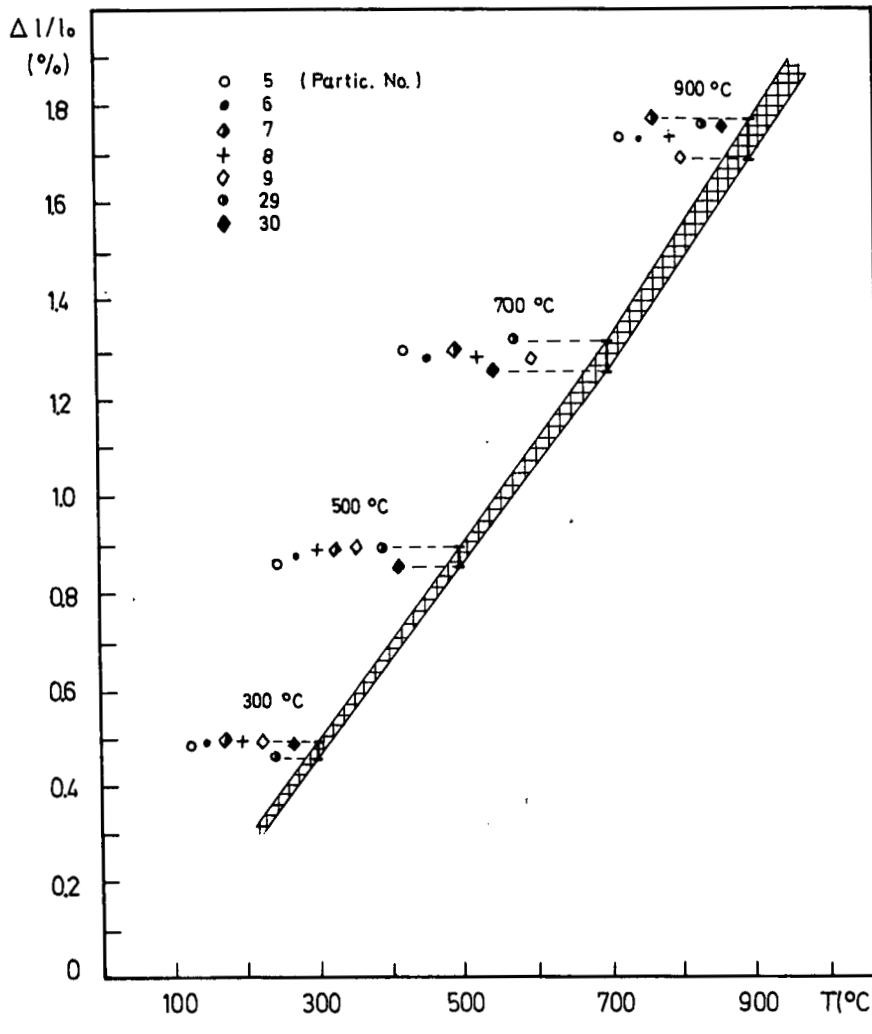

Fig. 16 Group scatter of the austenitic alloy after calibration with gold

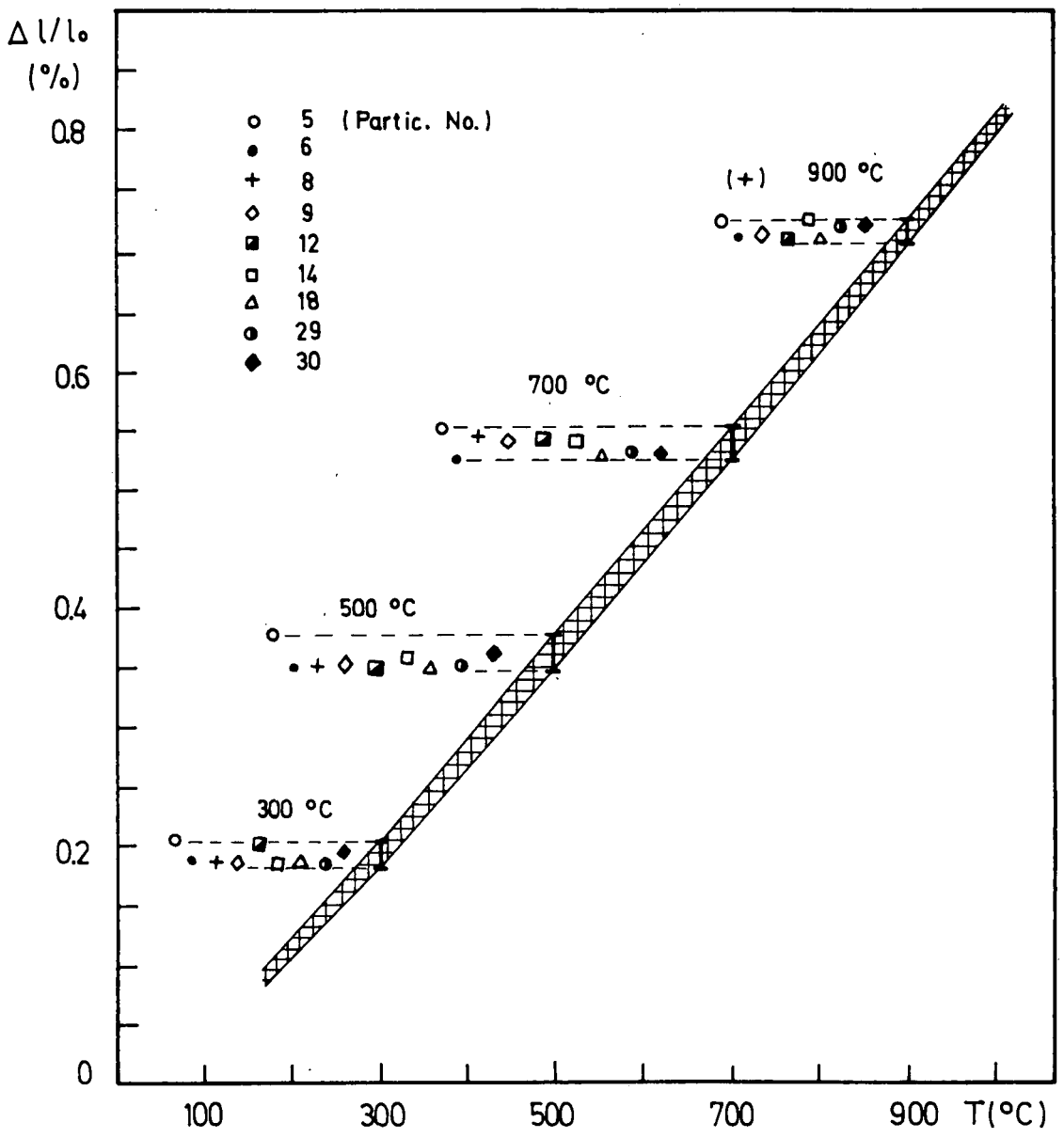

Fig.17 Group scatter of alumina

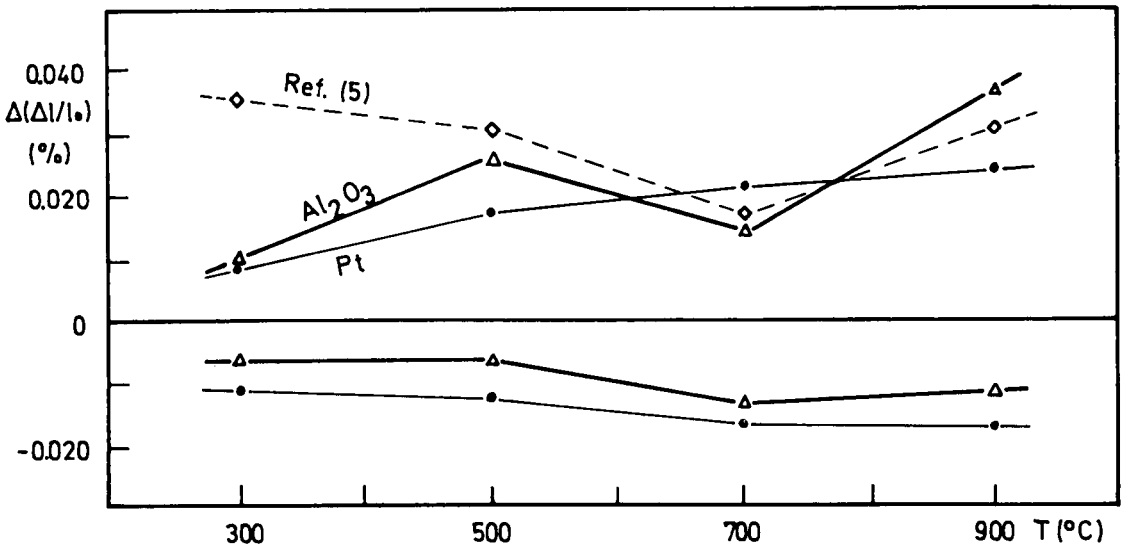

Fig.18 Group scatter for Al_2O_3 and Pt. The zero line represents the group mean. For comparison the deviation of known mean values (reported in Ref.(5)) from the group mean are also depicted

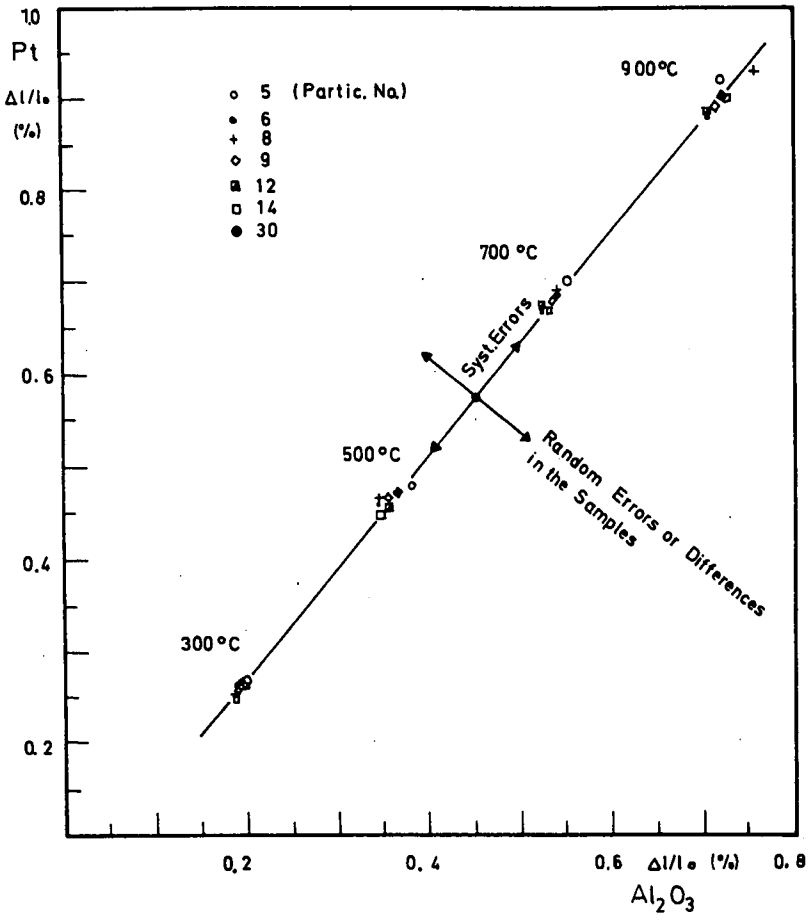

Fig.19 Error analysis plot Al_2O_3 versus Pt

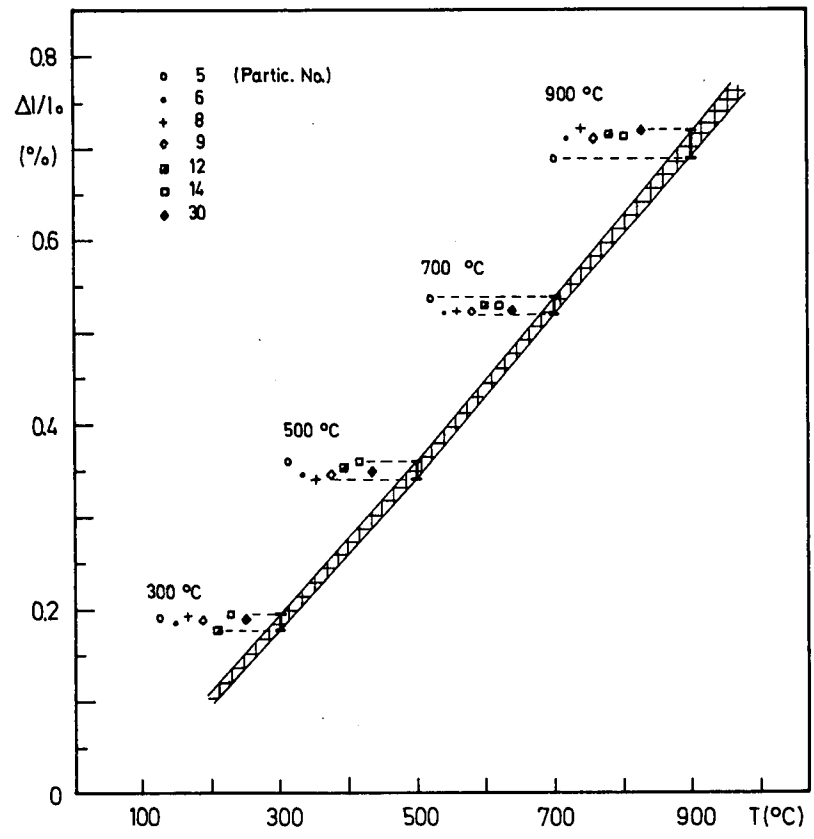

Fig.20 Group scatter for Al_2O_3 after calibration with Pt

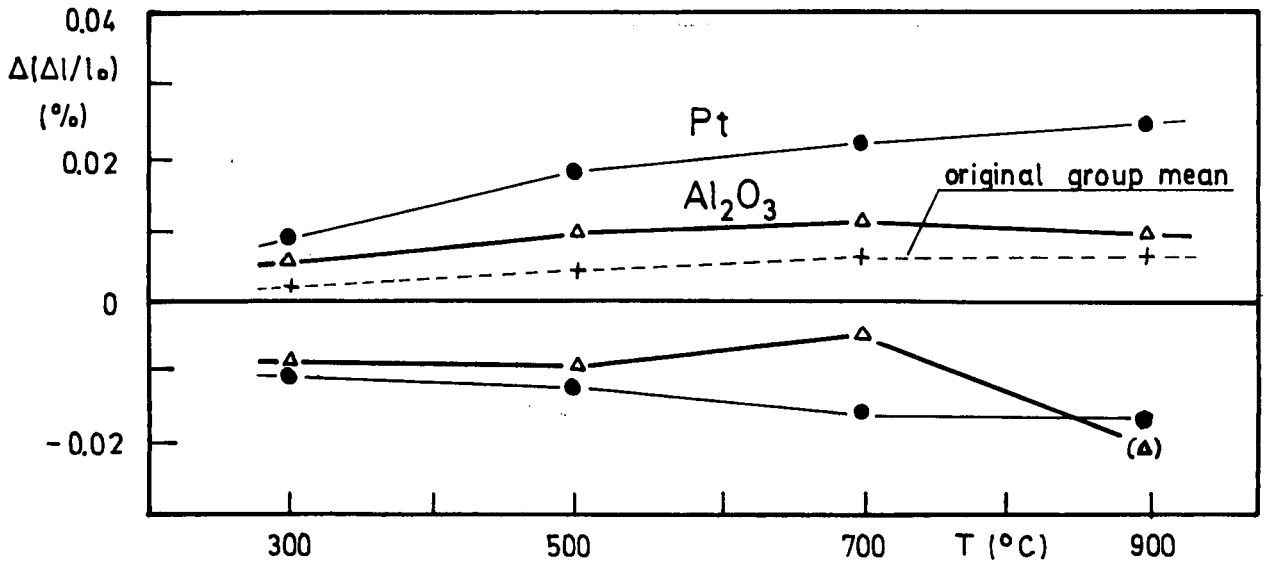

Fig.21 Group scatter obtained for Al_2O_3 after calibration with Pt, compared with the group scatter obtained for Pt. The zero line represents the new group mean for Al_2O_3. The original group mean for Al_2O_3 is included for comparison

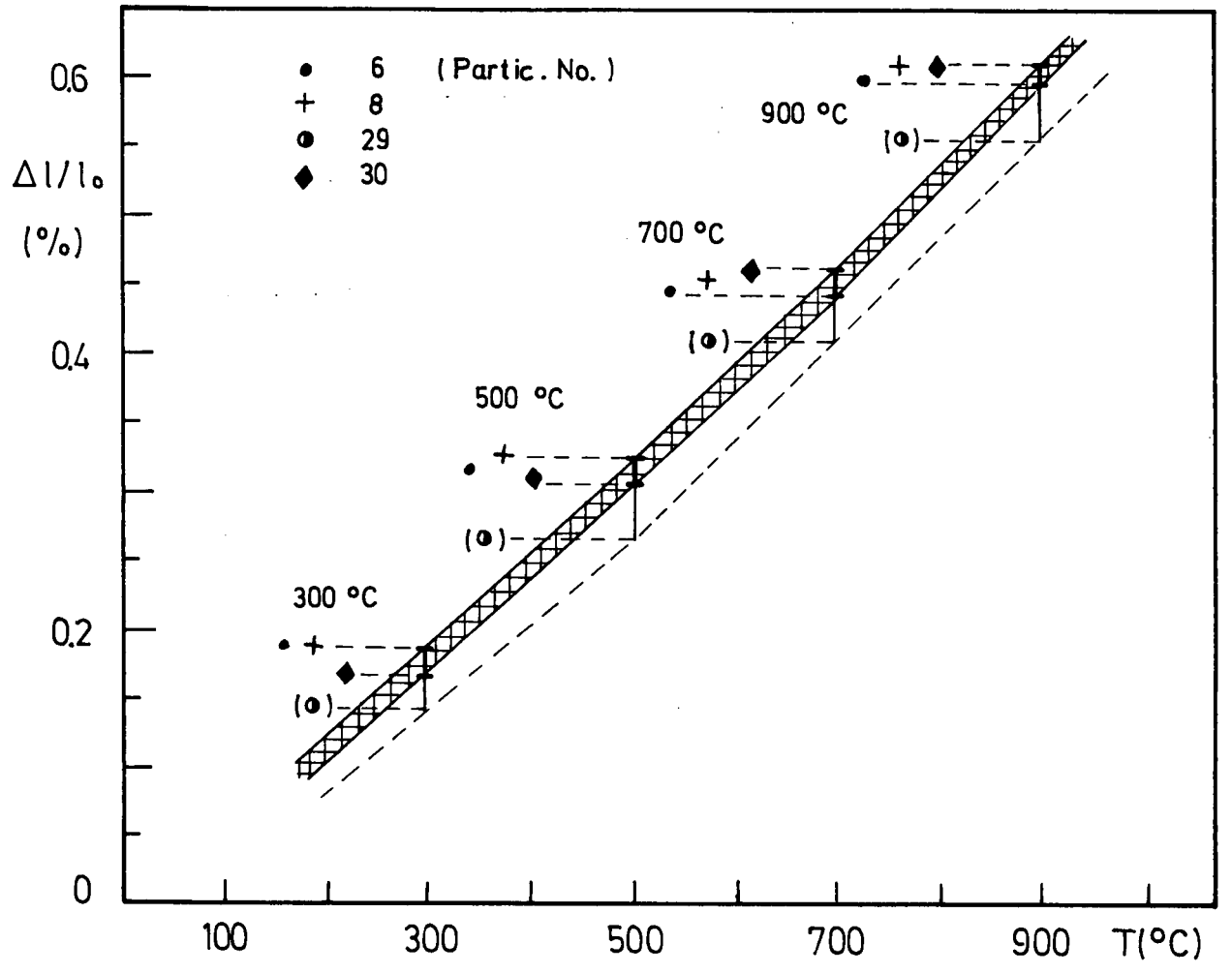

Fig.22 Group scatter of the individual means for Ta/10W

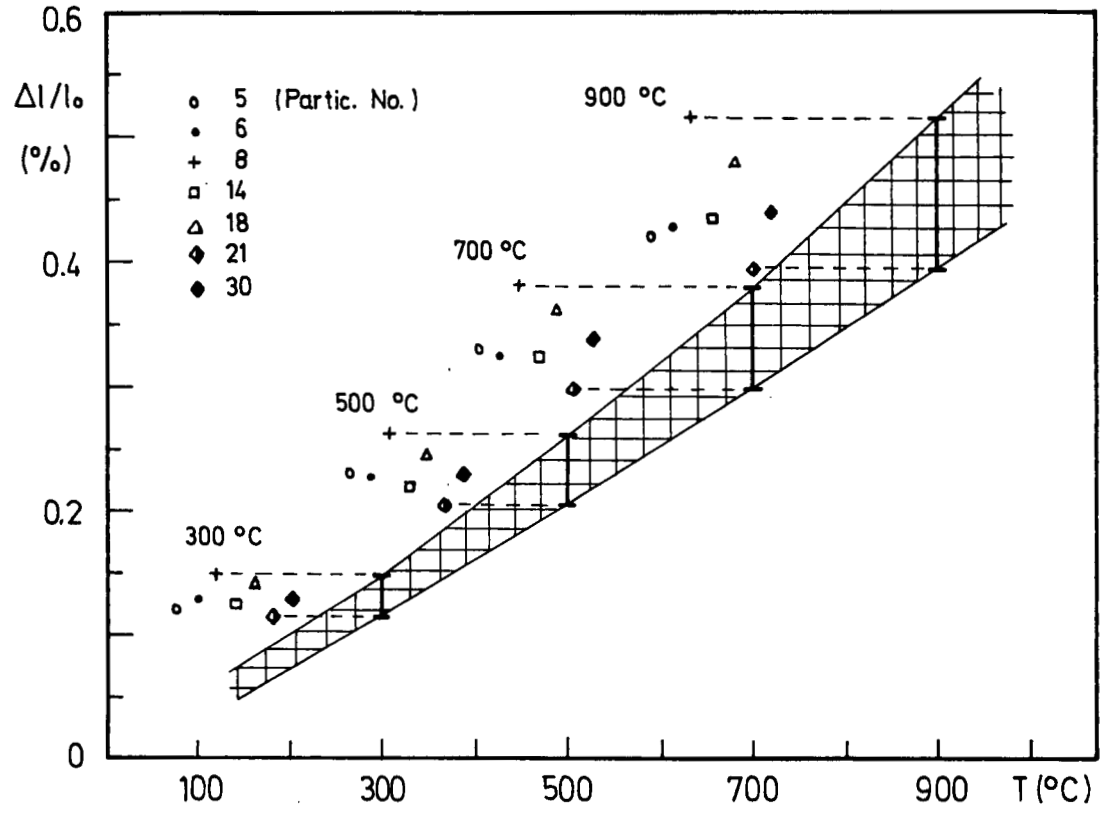

Fig.23 Group scatter of the individual means for the sintered tungsten samples

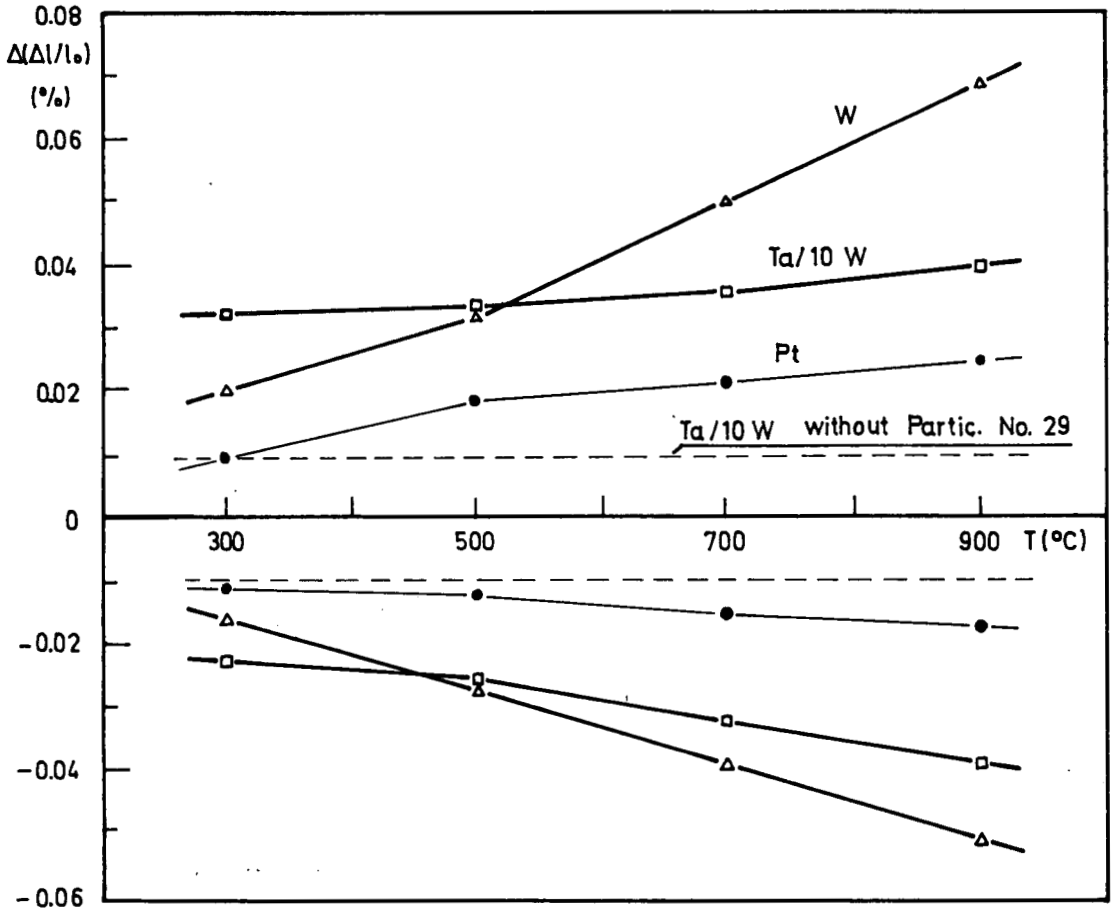

Fig.24 Deviation band of the individual means from the group average for Ta/10 W and sintered tungsten prior to calibration as compared to Ta/10 W after calibration and to Pt

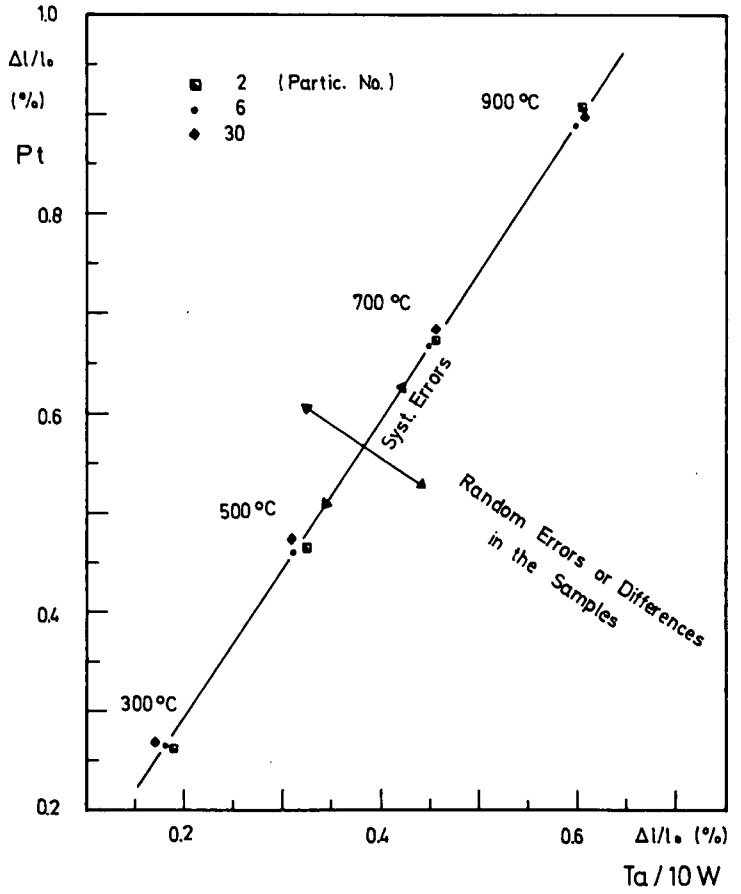

Fig.25 Error analysis plot Ta/10 W versus Pt

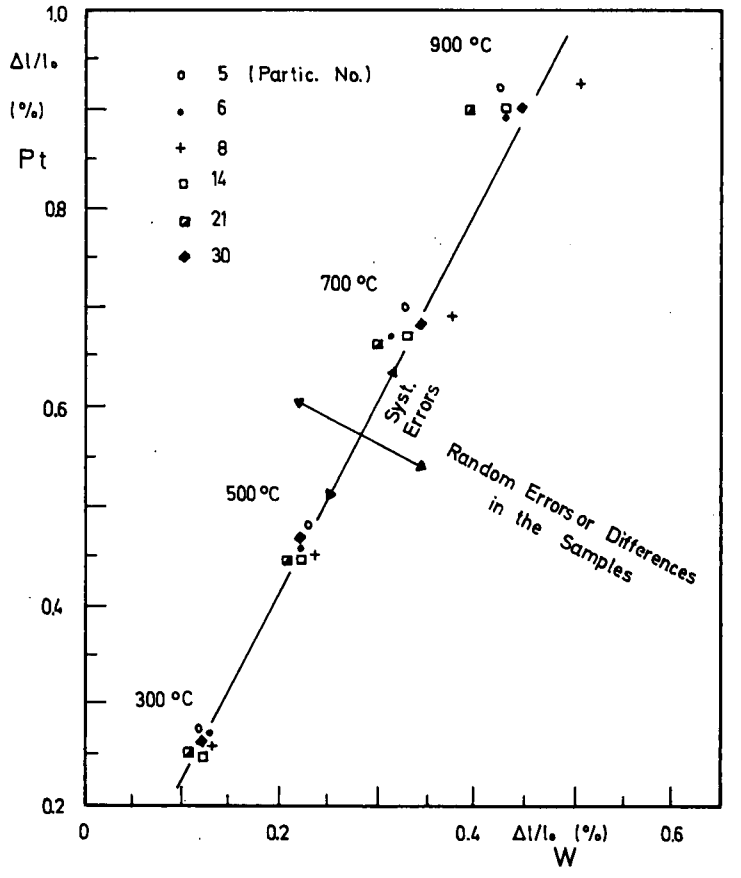

Fig.26 Error analysis plot W versus Pt

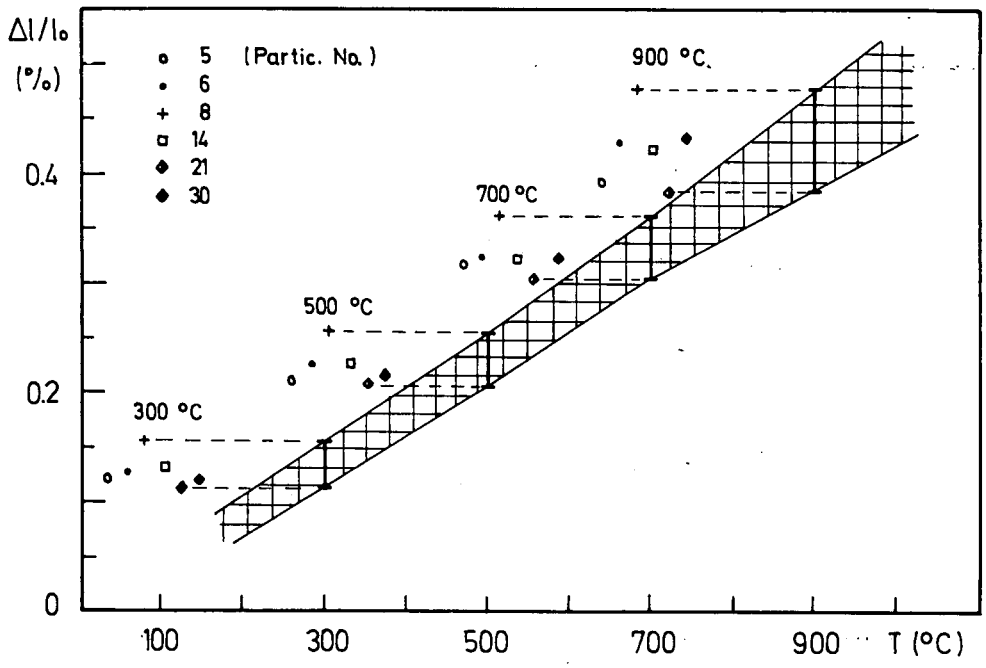

Fig.27 Group scatter of W-values after correction by calibration with Pt

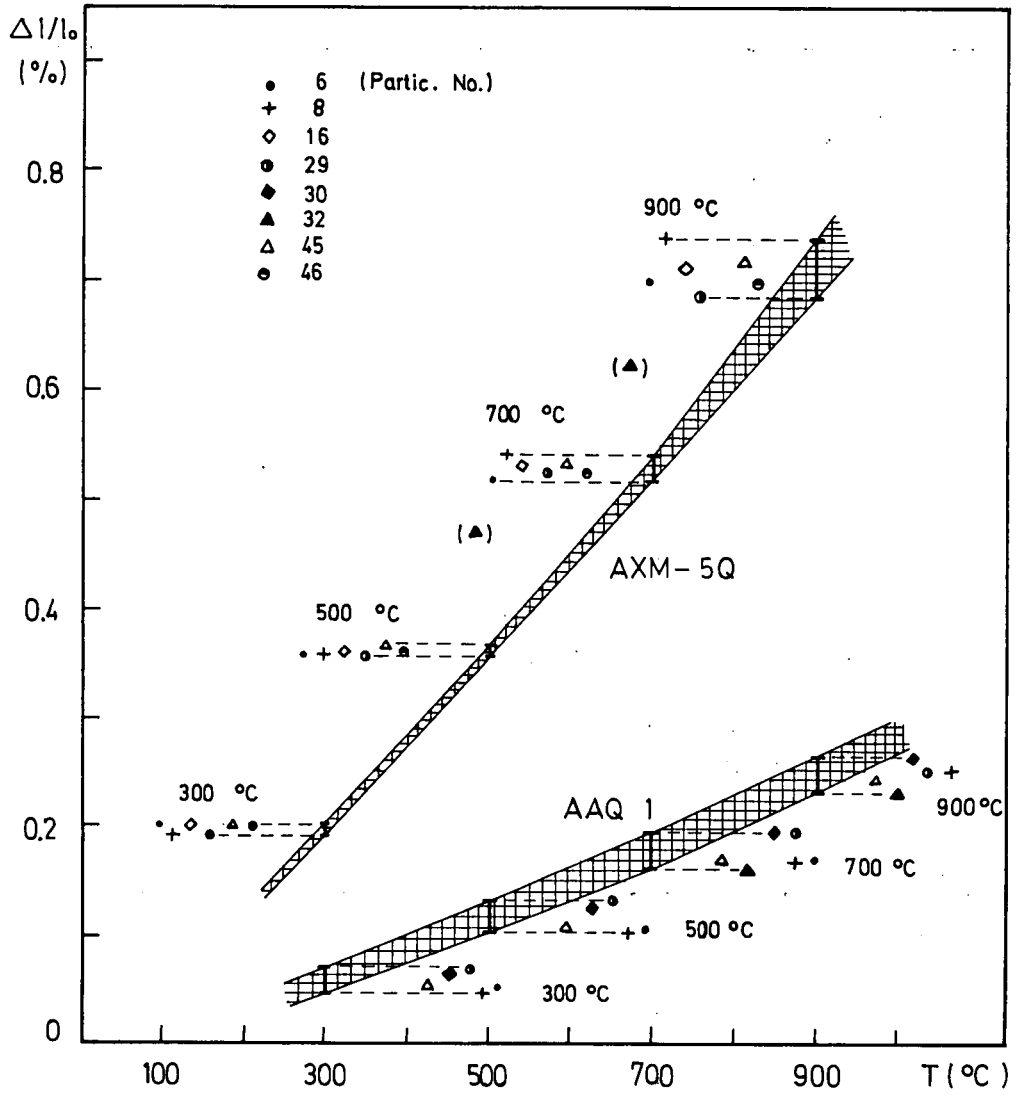

Fig.28 Group scatter of AXM-5Q and AAQ1-graphite

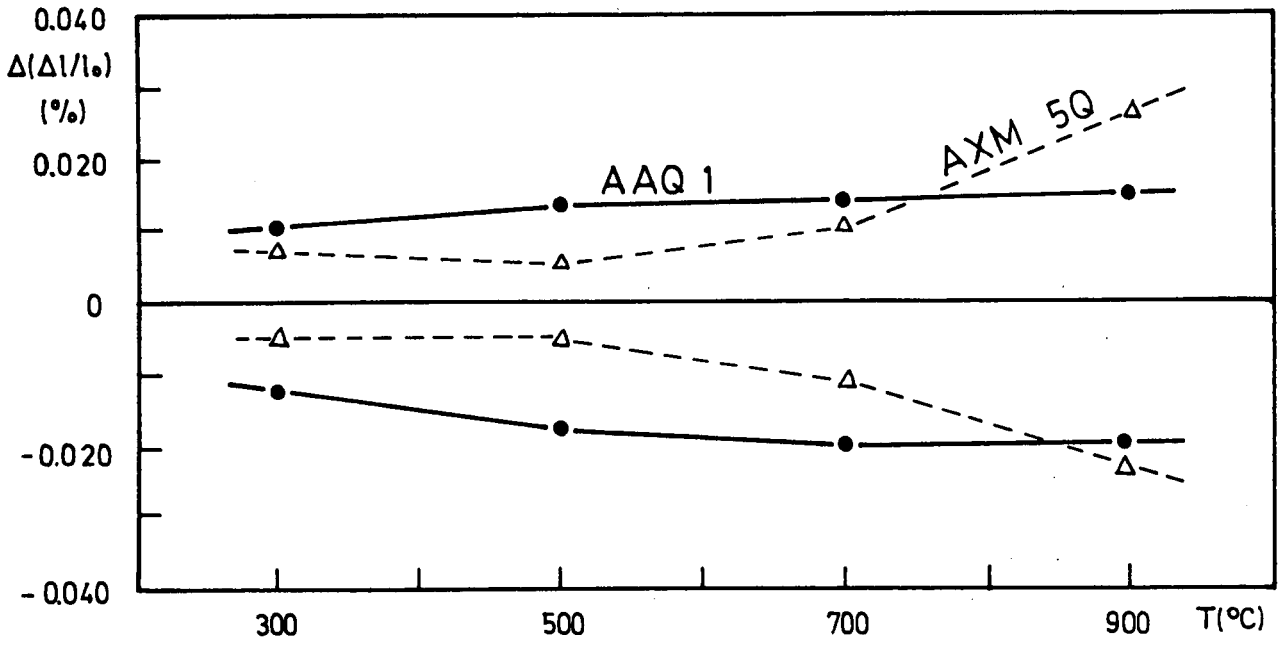

Fig.29 Deviation band of AXM-5Q and of AAQ1-graphite

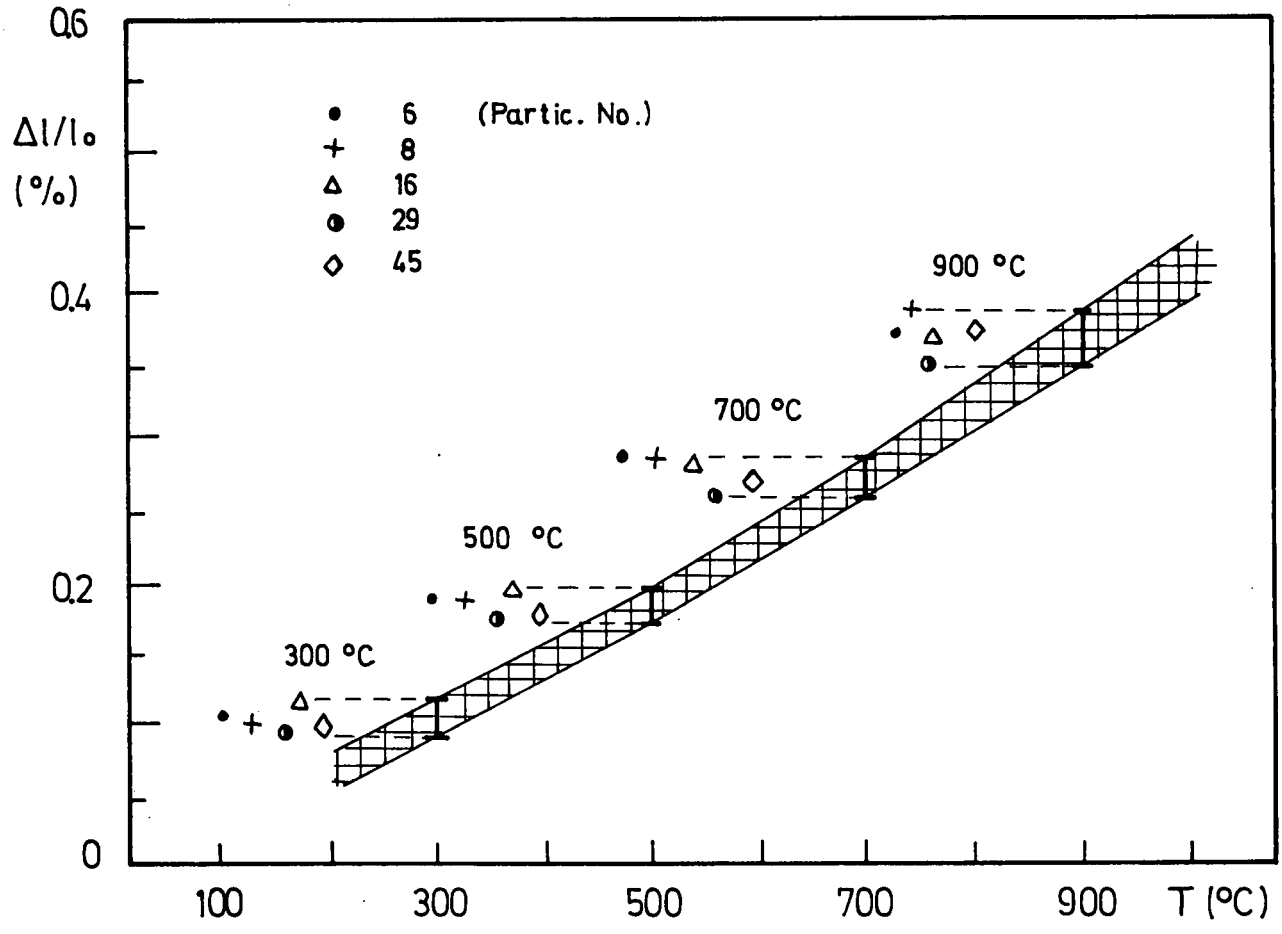

Fig.30 Group scatter of RVD-graphite with grain

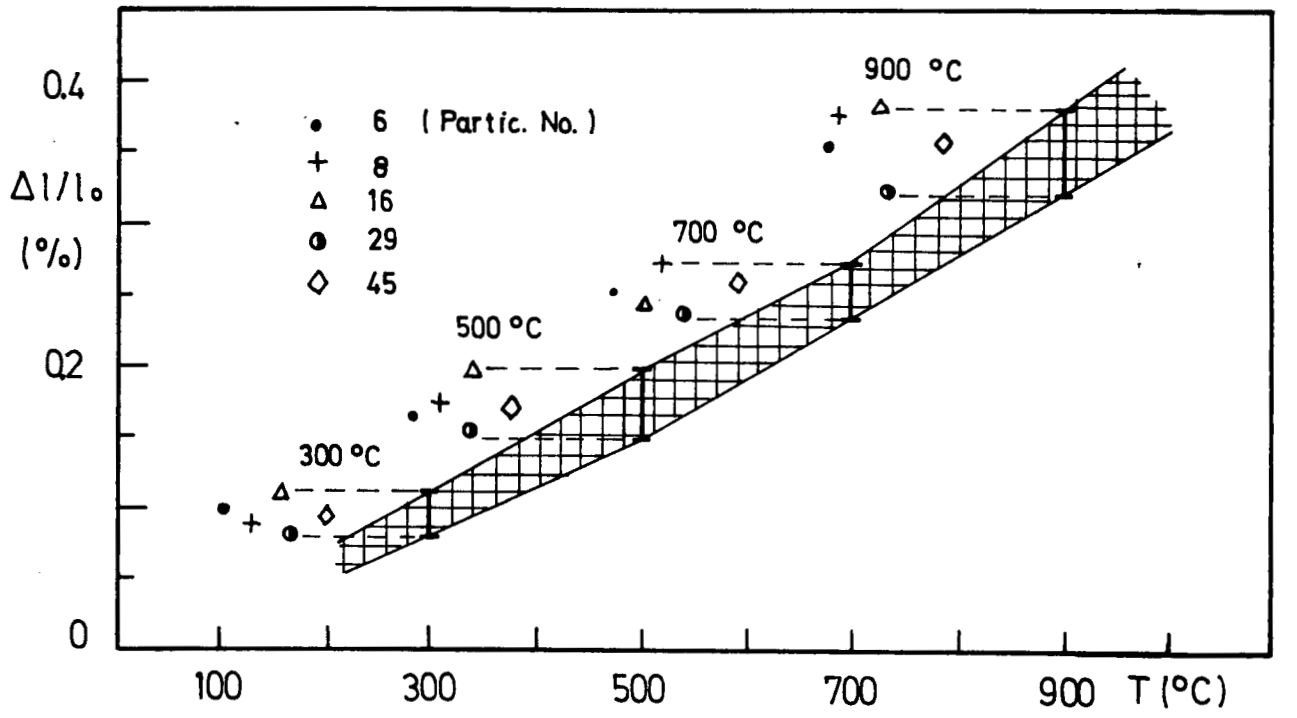

Fig.31 Group scatter pf RVD-graphite across grain

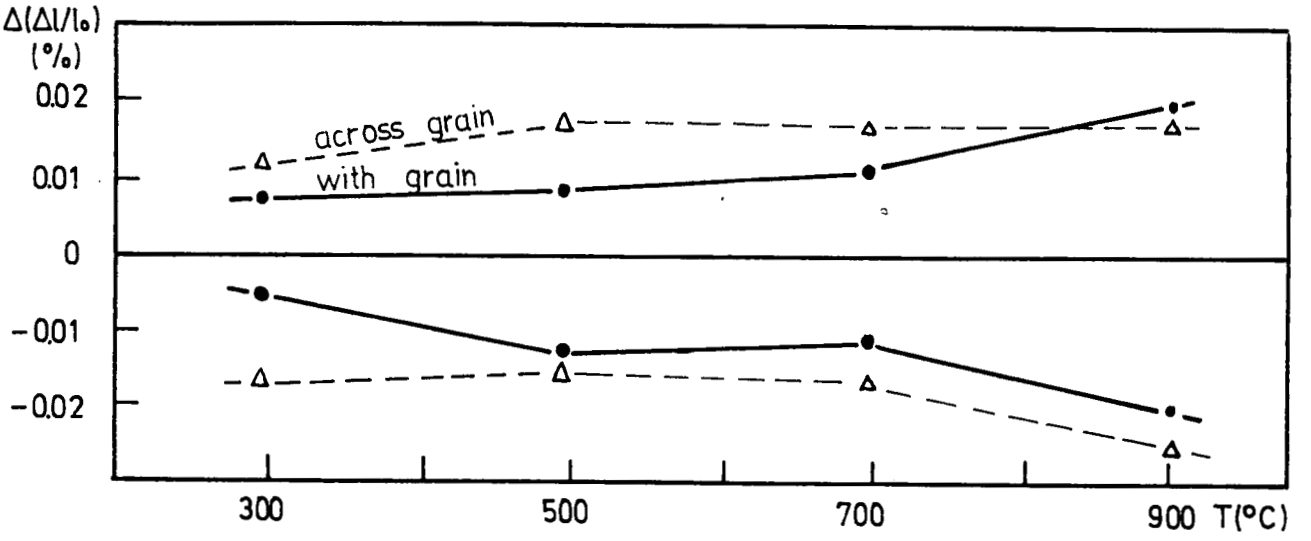

Fig.32 Deviation bands of RVD-graphite

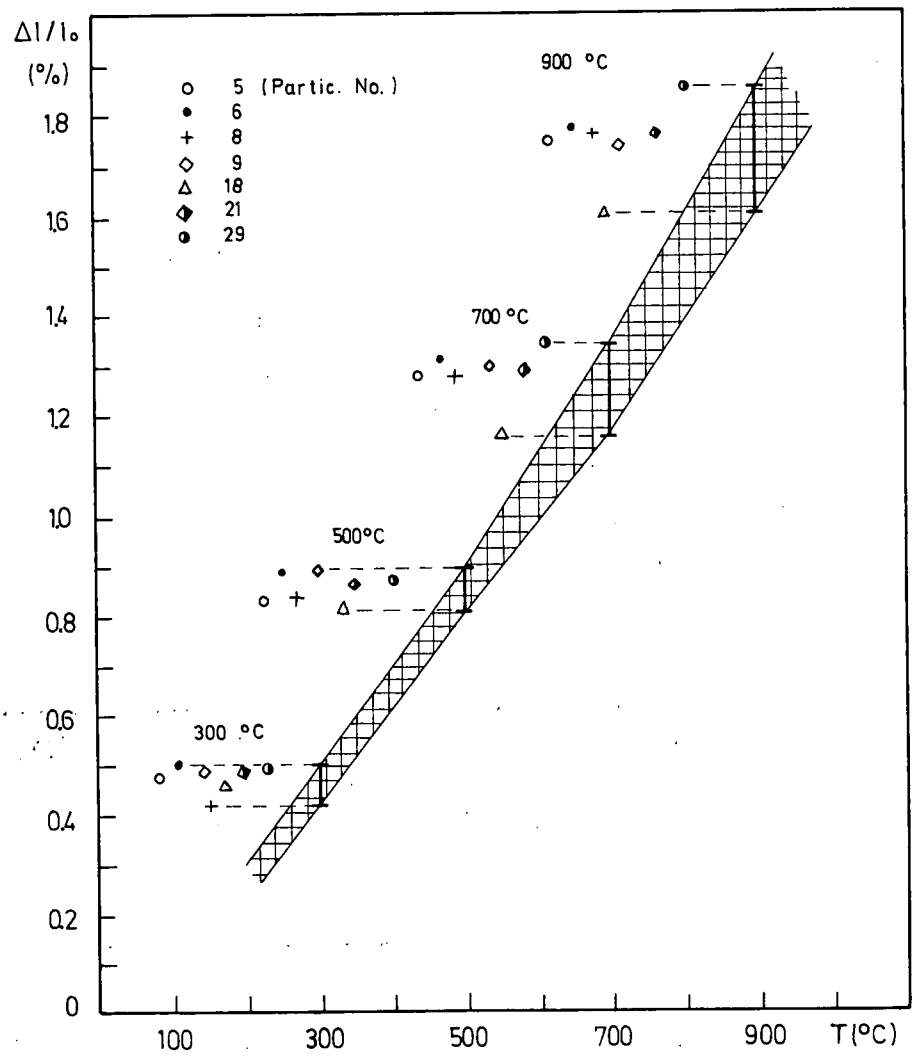

Fig.33 Group scatter of copper

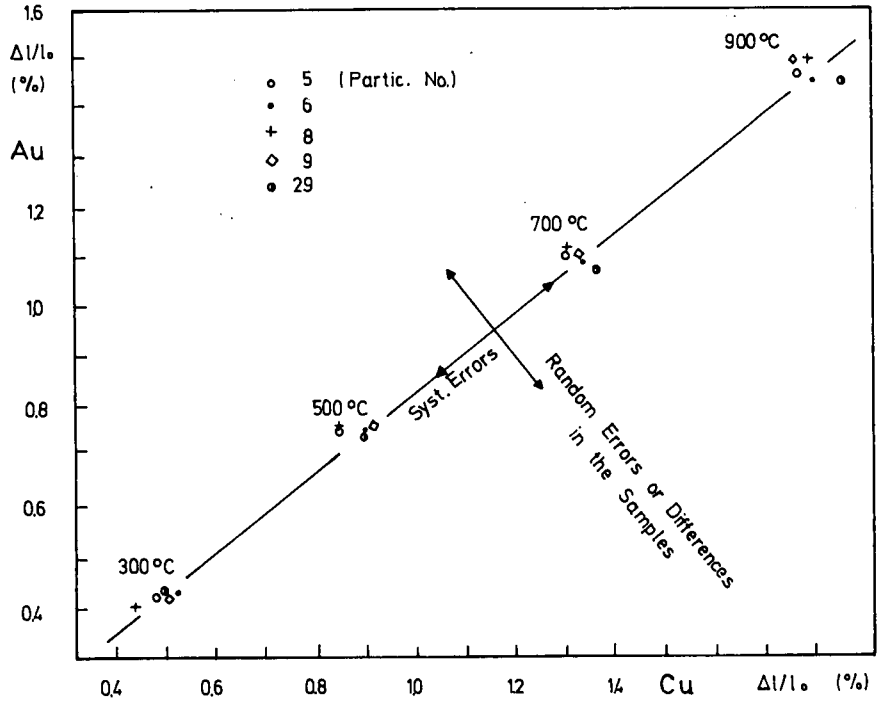

Fig.34 Error analysis plot copper versus gold

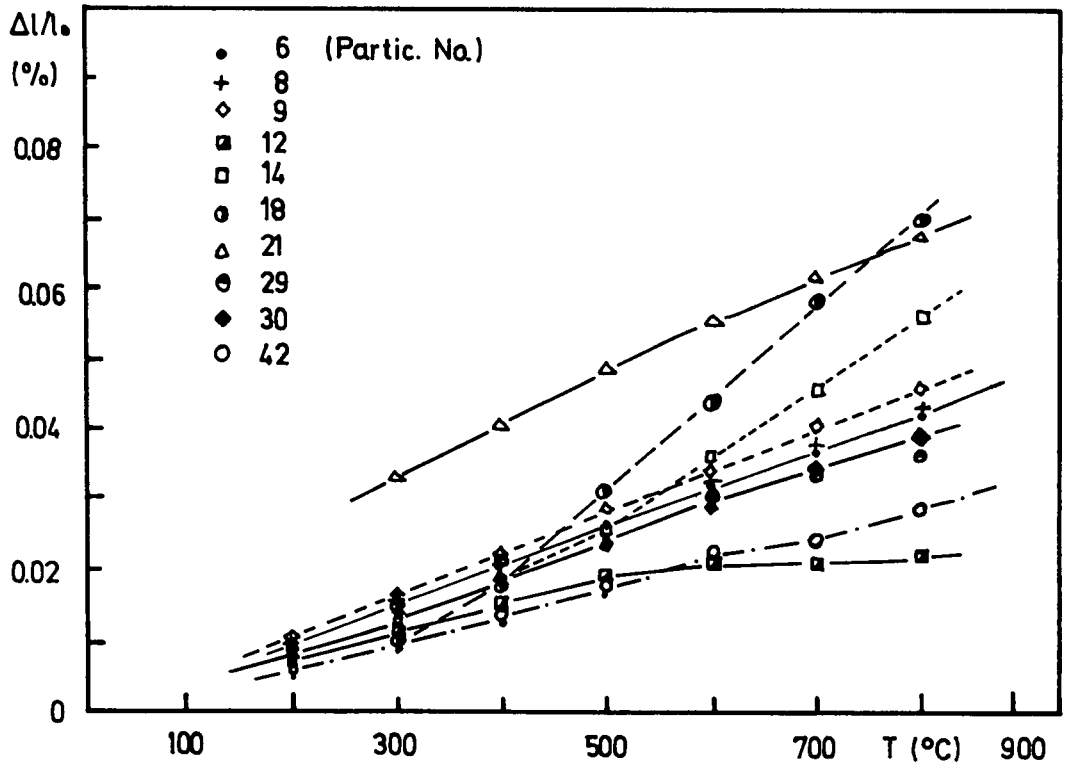

Fig.35 Group scatter of silica glass

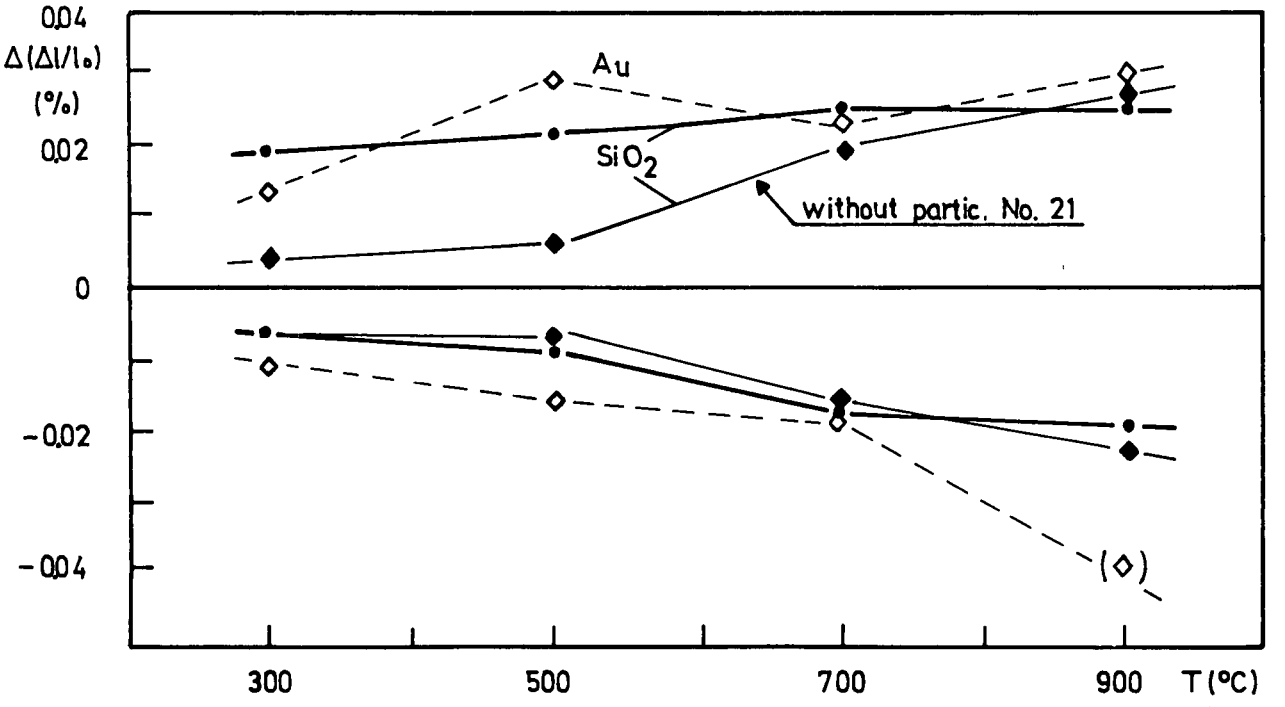

Fig.36 Deviation band of silica glass, compared with that of gold. The zero line represents the group mean

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Using high purity gold and platinum as selected reference materials, it was found that the relative pushrod dilatometer measurements when used in a group effort can yield an accuracy equal to that obtained by individual absolute measurements. This result was confirmed with engineering materials such as an austenitic alloy, a Ta/10 W alloy, sintered alumina and fine-grain graphites. This study is being extended to the highest possible temperatures.

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