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Elevated Temperature Modulus Measurements for Discontinuously Reinforced Metal Matrix Composites

This Measurement Note deals with the measurement of Young's modulus of discontinously reinforced metal matrix composites (MMC) between room temperature and 300°C. Results are presented which show the variation of modulus with temperature for a variety of MMCs, and a comparison of data from a VAMAS international round robin exercise on a whisker reinforced aluminium MMC.

A number of techniques were studied including dynamic modulus methods, the use of high temperature strain gauges and the application of a prototype capacitance gauge extensometer. Dynamic modulus measurements and strain gauge results gave good agreement from room temperature to ~200°C, and the fall in modulus over this temperature range is small, typically only a 6-8% reduction in the room temperature value.

One of the main goals of the work was to develop a test procedure for measuring modulus at elevated temperatures. A new test procedure has been developed, which is designed to be used separately from the conventional tensile test, but further work is still required to fully validate the method. Details are given in a separate Measurement Note. It is planned that the method will be submitted to ISO as an annexe to the ISO/TTA2 document on the tensile testing of MMCs

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Introduction

Many of the target applications for MMC seek to exploit the improved elevated temperature properties - particularly the specific strength and stiffness (modulus) - of the composite compared to the unreinforced matrix alloy. Because these parameters can be key factors in material selection, it is vital that accurate measurements of modulus and elevated temperature properties be made to give a true comparison of material performance. Some of the current applications for discontinuously reinforced MMCs include automotive brake parts and engine components (piston crowns, cylinder liners, connecting rods etc.), structural parts in aircraft, electronic instrument housings, general engineering applications and leisure goods. Typical operating temperatures for a selection of components are given in Table 1. If modulus is the major design parameter for a particular application of MMC, specific attention should be taken regarding the test methodology because the proportional limit and range of elastic behaviour can be very small, and this places the emphasis on accurate strain measurement.

Standards

At present there are no standards for measuring the Young's modulus of MMCs and even the methods for the tensile testing of metals are vague in their recommendations regarding modulus measurement. ASTM E111-97 [1] is the only standard currently available which deals specifically with the measurement of modulus from mechanical tests. The European equivalent, EN 10002 [2], which has five separate parts, deals with various aspects of the tensile test including the calibration of the test machine and extensometry, but does not provide any explicit instructions regarding the measurement of Young's modulus itself. ASTM E1875-97 [3] and ASTM E1876-97 [4] cover dynamic modulus methods.

Previous work on MMCs at NPL [5-7] has highlighted the importance of using double-sided strain measurement for accurate modulus values at room temperature, and it is reasonable to assume that a similar practice should be used at elevated temperatures. Both ASTM E111-97 and EN 10002 state that averaging extensometry is preferred but not mandatory, and the only recommendation for elevated temperature tensile testing in EN 10002-Pt5 is that a Class 1 extensometer be used [2]. From the work on MMCs, strain measurement methods and data analysis techniques are probably the two most important issues which effect the accuracy and reliability of the modulus measurements. Specific guidelines on using double-sided strain measurement methods together with recommendations on data analysis were proposed in a draft procedure, which has since been accepted as an ISO TTA document [8].

Dynamic Modulus Methods

A variety of dynamic modulus methods are available including:

- Flexural resonance methods
- Ultrasonic wave propagation
- DMTA

The most commonly used methods for metals and ceramics are the resonance techniques. The dynamic methods are relatively quick and simple and involve very small elastic strains and high strain rates. Some can be readily modified to enable high temperature measurements. In the present work at NPL, tests have been carried out on a variety of MMCs using the Impulse Excitation Technique (IET), which is described in some detail below.

Impulse Excitation Technique

The Impulse Excitation Technique (IET) is a dynamic modulus method, which involves the excitation of a testpiece by a light mechanical impulse and subsequent analysis of the fundamental resonant frequency of flexural vibration. The testpieces were supported at the nodes of resonance in a furnace and struck by small ruby balls dropped down a guide tube to hit the centre of the testpiece. The fundamental resonant frequency is measured via a piezo-electric transducer in contact with the specimen. Further details on the test methodology are given in Refs 9-11.

The equation used to calculate modulus is:

$$E = 0.9465 \frac{m}{w} f^2 \left(\frac{L}{t} \right)^3 T_1$$

where:

m = mass of the testpiece
w = width
L = length
t = thickness
f = fundamental resonant frequency for flexural vibration

and T_1 is a shape factor, which for a long, thin rectangular bar with $L/t \geq 20$, can be approximated to:

$$T_1 = 1 + 6.585 \left(\frac{t}{L} \right)^2$$

The mass and dimensions of the specimens were measured prior to testing and measurements of the resonant frequency were taken at 25°C intervals during both the heating and cooling cycle. The testpiece was held at temperature for 10 minutes prior to measurement to ensure equilibrium and temperature uniformity. At least two tests were carried out at each temperature interval and the mean frequency value used to calculate the elastic modulus.

The results from a series of tests carried out using the IET are plotted in Fig 1a to show the variation of modulus with temperature and in Fig 1b, normalised with respect to the room temperature modulus. The calculations of modulus were made using the formulae above, assuming isotropic behaviour and with Poisson's ratio μ = 0.25.

The MMCs and matrix materials show a fairly steady fall in modulus with increasing temperature. Over the same temperature range the magnitude of the reduction is greater in the MMCs compared to the unreinforced matrix materials, but the normalised data shows a remarkably similar behaviour for all the materials examined. Closer inspection of the data shows that the variation of modulus with temperature is non-linear, and the values begin to fall more rapidly above a temperature of 200-250°C.

An important limitation of the IET is the existence of a fairly well defined temperature limit above which

the fundamental frequency signal cannot be identified. It is not clear what causes this, but the behaviour has been ascribed to internal friction, plasticity and damping. It is difficult to predict the exact temperature at which this will occur because the temperature limit is material specific and does not seem to be affected by the test setup or specimen geometry. In the present work the practical limit was about 425°C for the aluminium matrix materials and between 325-350°C for the MMCs. The approach of the temperature limit can be noted on the frequency analyser display as a broadening in the peak of the fundamental frequency accompanied by a sharp reduction in amplitude.

A further consideration is that the technique is only suitable for isotropic, homogeneous materials. Particulate reinforced MMCs seem to satisfy these criteria, but additional tests on fibre reinforced MMCs were unsuccessful, because it was not possible to obtain a consistent resonant frequency response.

Static Modulus Methods

One of the main problems associated with measuring modulus during the tensile testing of MMCs arises from the low proportional limit and limited range of elastic behaviour. For example, the elastic behaviour of the MMC examined in this work is limited to ~ 0.1% strain at room temperature, and this is reduced to less than 0.04% at 225°C. Because of the restricted elastic strain range, there is considerable emphasis on accurate strain measurement, yet it is unrealistic to expect extensometers or other strain measuring devices which have been calibrated to measure strains of up to 5% or more, to have sufficient resolution to measure modulus accurately over this limited strain range (for an extensometer with a 25mm gauge length, 0.1% strain equates to a deformation of only 0.025 mm).

Table 1: Typical applications of discontinuously reinforced MMC.

Target Application	**Operating Temperature**	**Property Requirements**
Automotive brake parts	-20 to 300°C	High specific stiffness, strength, creep, wear resistance
Automotive engine parts	0 to 400°C	Elevated temperature strength and stiffness, wear resistance
Aircraft engine structures, helicopter gearbox casing	-20 to 200°C	High specific stiffness, strength, creep and damage tolerance
Aircraft fuselage	-50 to 200°C	Creep resistance, strength and stiffness
Electronic instrument housings, circuit board	-70 to 200°C	Low mass, stiffness, tailored CTE

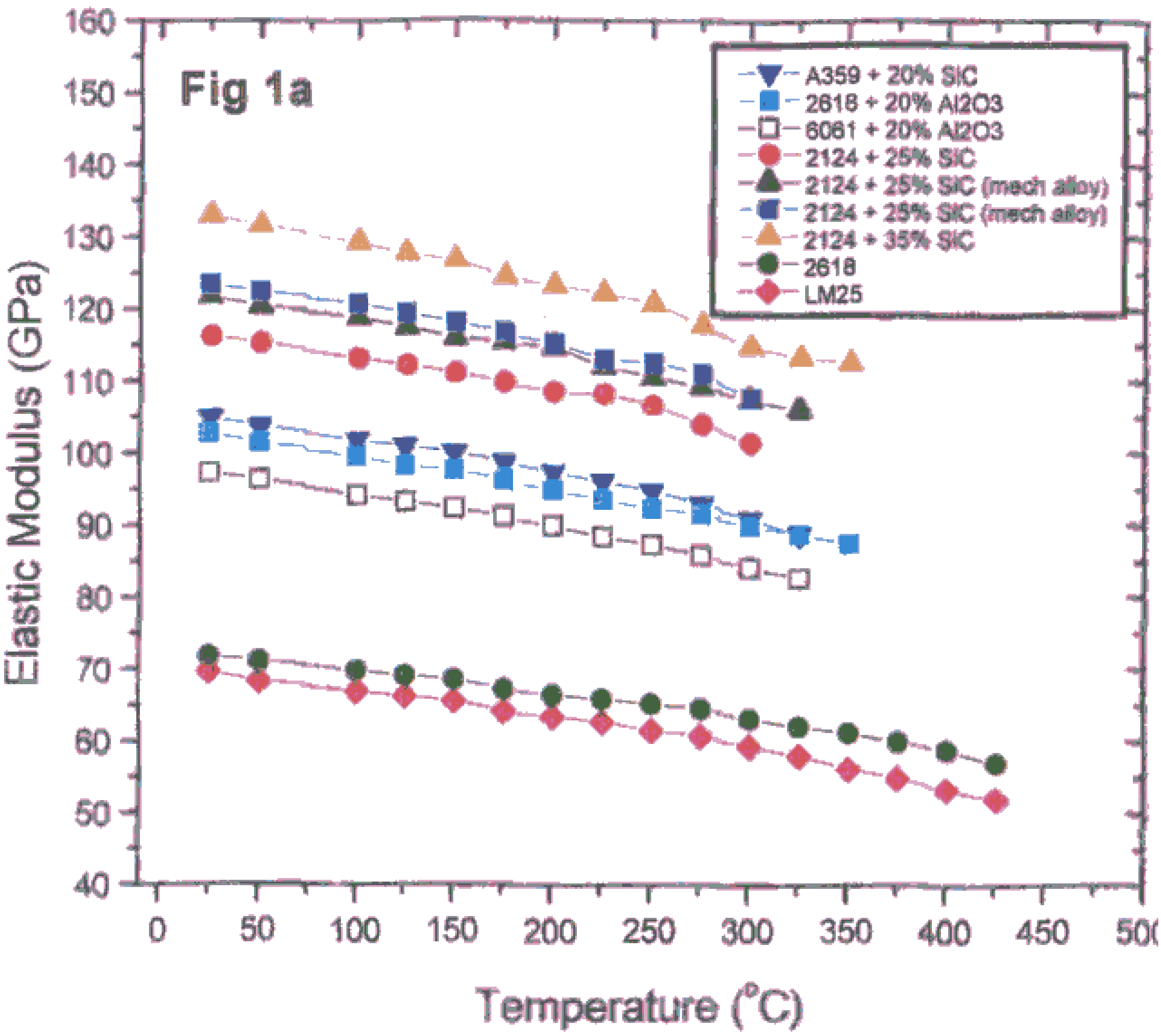

Figure 1: Variation of modulus with temperature for a variety of MMCs and matrix materials (IET data)

Strain Gauges

High temperature strain gauges are available from most strain gauge suppliers. In general they can be used in the same way as the room temperature varieties, but preparation and installation is more demanding. Conventional Cu-Ni foil gauges which use epoxy or polyester backings typically have a maximum operating temperature of 80° C; high temperature variants which employ Ni-Cr foil and polyimide backings can be used up to 300°C, and welded gauges up to 650°C.

If high temperature strain gauges are used a number of practical points must be considered, including the use of :

- high temperature adhesives
- high temperature solder
- coated leadwires
- protective coatings for gauges
- matched CTE

Ideally the gauges should be chosen to have similar thermal expansion characteristics to the testpiece, to minimize thermal effects. Care must be taken to allow the gauges to reach an equilibrium temperature and they should be balanced immediately prior to testing as heating can lead to the generation of large thermal strains. An important point for many MMCs and other heat treatable alloys is that in many cases it is not possible to use an adhesive which cures at high temperature because this may lead to the material overaging which affects the mechanical properties.

A series of tests were carried out to measure the variation of modulus with temperature of three representative materials - 2 MMCs and a 2618 aluminium alloy, during conventional tensile tests using strain gauges. High temperature gauges were bonded to both sides of a rectangular dogbone testpiece using a cold curing epoxy adhesive. High temperature solder and PTFE coated leadwires were also used and the whole gauge installation was coated with a silicon rubber compound for additional protection. The temperature of the specimen was monitored during the test using a type R thermocouple attached to the gauge length.

For each material a single specimen was used over the whole temperature range, loaded within the elastic regime, with measurements at 25°C intervals. The testpiece was loaded at each temperature from 0-2 kN at a crosshead speed of 0.5 mm/min, and only at the higher temperatures (above ~200°C) was there any indication of plastic deformation during the loading cycle. The gauges were balanced after heating to the next temperature step andimmediately prior to testing to eliminate any thermal strains generated during heating.

Young's modulus was calculated from the stress-strain data using the analysis procedure developed at NPL [Method M3 in Ref 8] which examines the best fit to the secant and tangent moduli to give an accurate fit to E.

The variation of modulus over the temperature range examined is shown in Fig. 2 for the relevant materials and a Nimonic 901 specimen, together with the IET data for comparison.

Up to ~200°C there is reasonable agreement between the data from the IET and strain gauge tests. Above this temperature however, the strain gauge tests gave lower values and the difference gets larger as the temperature increased. Results for the Nimonic specimen showed reasonable agreement for the two methods up to 300°C which suggests that the integrity of the gauge and adhesive is still intact. (The gauges themselves were rated to 300°C, and the adhesive from -30 to 300°C) The difference between the IET data and the strain gauge results for the MMC above 200°C is difficult to explain, particularly when the results for the Nimonic 901 are in reasonable agreement up to 300°C. One possible explanation could result from the larger strains experienced in the aluminium-based materials during the loading cycle compared to the Nimonic alloy, which may lead to local debonding, plastic deformation or creep within the adhesive layer. This would lead to larger strains and lower modulus values. Results from tests carried out using a cyanoacrylate adhesive, which is designed to operate at a maximum temperature of 120°C, gave consistent values up to this temperature limit but debonded at higher temperatures.

A stable adhesive should be used, with the general recommendation that the adhesive is cured at least 20°C above the maximum temperature encountered in the test, but this is not always possible with MMC, particularly if the material is heat treatable. The gauges and adhesive combination should be considered carefully, and any variation in gauge factor with temperature should be included in the strain measurement. The influence of thermally generated strains can be eliminated by balancing the gauges immediately prior to testing.

Clearly a large number of adhesive/material combinations exist and it is difficult to give definitive recommendations on their use. Further work is still required before definitive recommendations can be made regarding using strain gauges for measuring the Young's modulus of aluminium-based MMCs at temperatures above ~200°C.

Extensometers

A wide variety of extensometers exist for mechanical testing, including the conventional clip-gauge designs and the more sophisticated ceramic rod-based systems, but there is a lack of simple designs for double sided strain measurement at elevated temperatures. Extensometers based on strain gauge sensors are limited by the operating range of the gauges themselves, although water cooled systems are available, but this makes the design bulky. In the present work, although only moderate temperatures (200-300°C max.) are being considered, few commercial double-sided systems are available. Both EN 10002-Pt 5 and ASTM E111 recommend that double-sided averaging extensometers or two separate extensometers be used, but this is not a condition of the test.

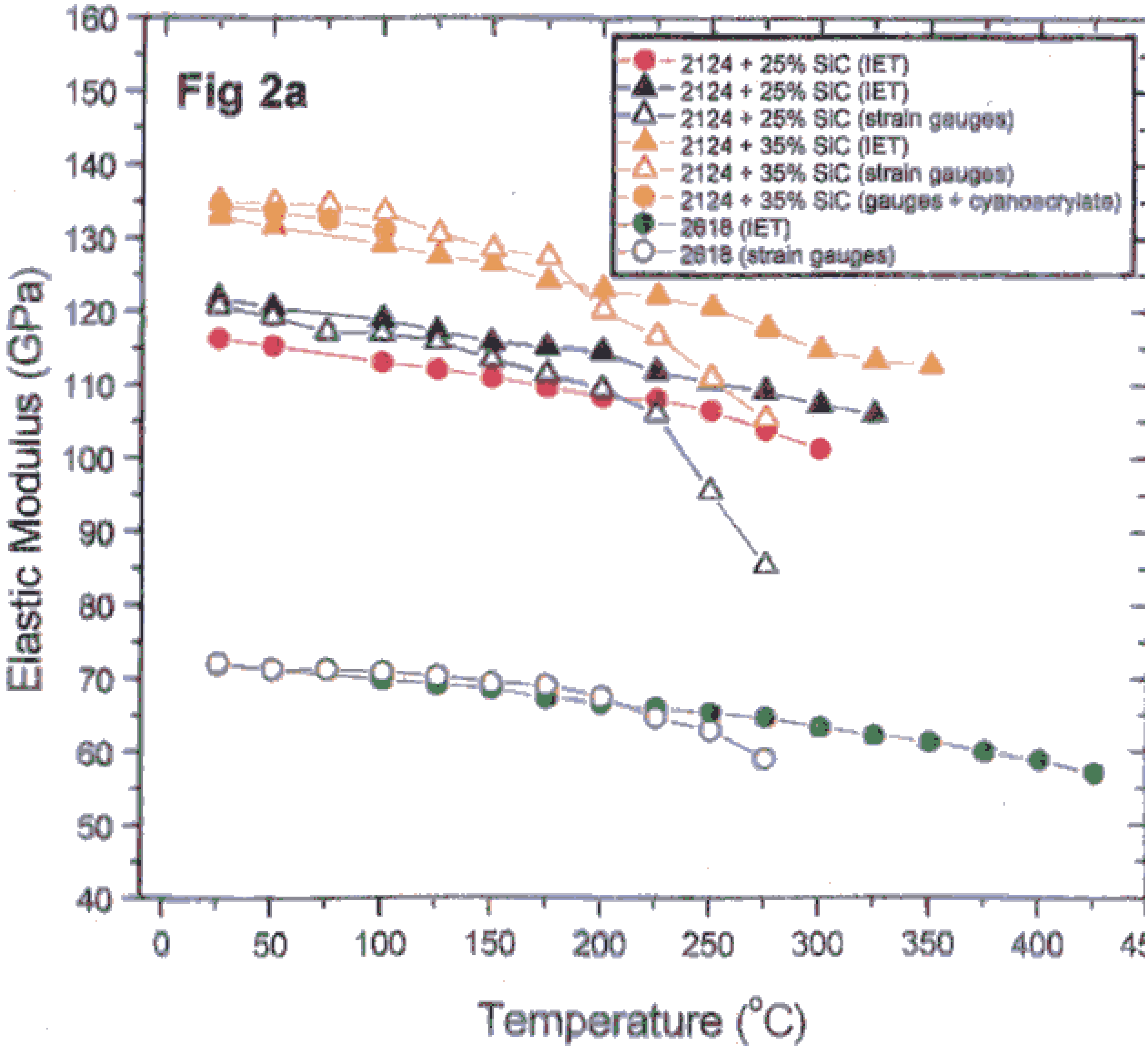

Figure 2: Young's modulus data from strain gauge tests for a variety of MMCs and matrix materials

The European Standard on uniaxial high temperature tensile testing, EN 10002-Pt 5, does not contain any specific reference to the measurement of Young's modulus, but simply specifies the use of a Class 1 extensometer for the measurement of upper and lower yield strength and proof strength. This implies a potential accuracy of ± 1% for the measurement of strain when testing to this standard, and this is approximately one-half the accuracy achievable when testing to the previously used BS 3688. In practice however, potential errors of between 10-20% in the measurement of strain can be expected over the limited elastic strain range being considered for MMCs and this effects the accuracy of the Young's modulus determination.

VAMAS Intercomparison Exercise

Tests have been carried out by NPL as part of two intercomparison exercises organised by VAMAS TWA15 to measure the elevated temperature tensile properties of a whisker reinforced MMC. The results from the first exercise are reported in Ref 12 and the repeat exercise in Ref 13. The tensile tests were carried out at 200°C and 3 different strain rates were used in the second exercise, following specific guidelines drawn up after the first study to try and reduce the scatter in the results - particularly proof stress and tensile strength. The guidelines included details on the heating profile and the time held at temperature prior to testing. The modulus results from both exercises are plotted in Fig 3, together with details on the methods used for strain measurement (E is used for extensometry, G for strain gauges; D refers to double-sided, S to single sided strain measurement) and for calculating Young's modulus, as described in Ref 8 and detailed below:

Method M1 is a graphical method and the modulus is calculated from the slope of the line drawn over the initial part of the stress/strain curve;

Method M2 is a chordal method, often used with computer software, and the modulus is calculated from the straight line joining two points on the stress/strain curve;

Method M3 uses a quadratic polynomial fit to the stress/strain curve and examination of the tangent and secant moduli to obtain a value for Young's modulus.

In the first exercise, the mean value for Young's modulus was 97.0 ± 2.7 GPa (excluding 2 outliers), and including all the data the value was 101.0 ± 5.5 GPa. In the repeat exercise the mean value for the modulus at 200°C was 98.6 ± 6.1 GPa (excluding 3 outliers) and 97.1 ± 13.0 GPa for all the data. At room temperature the Young's modulus is 104.5 GPa, so there is only a relatively small reduction in E as a result of heating to 200°C.

The data in Fig 3 shows that the uncertainty involved with measuring Young's modulus in the repeat exercise is higher than the first study, but it is not immediately clear why this is the case. One reason for the increased scatter could be that a number of new organisations were involved in the second exercise and they were not familiar with the test procedure. An additional recommendation for this series of tests was to put the testpiece into the test machine at the test temperature. In hindsight this may have caused increased scatter in the data because of the practical problems of alignment and positioning of the testpiece in the grips at temperature. It is interesting to note that significant reductions in the scatter associated with the proof stress and strength measurements were achieved by specifying more closely the heating rate and dwell times at temperature prior to testing [13].

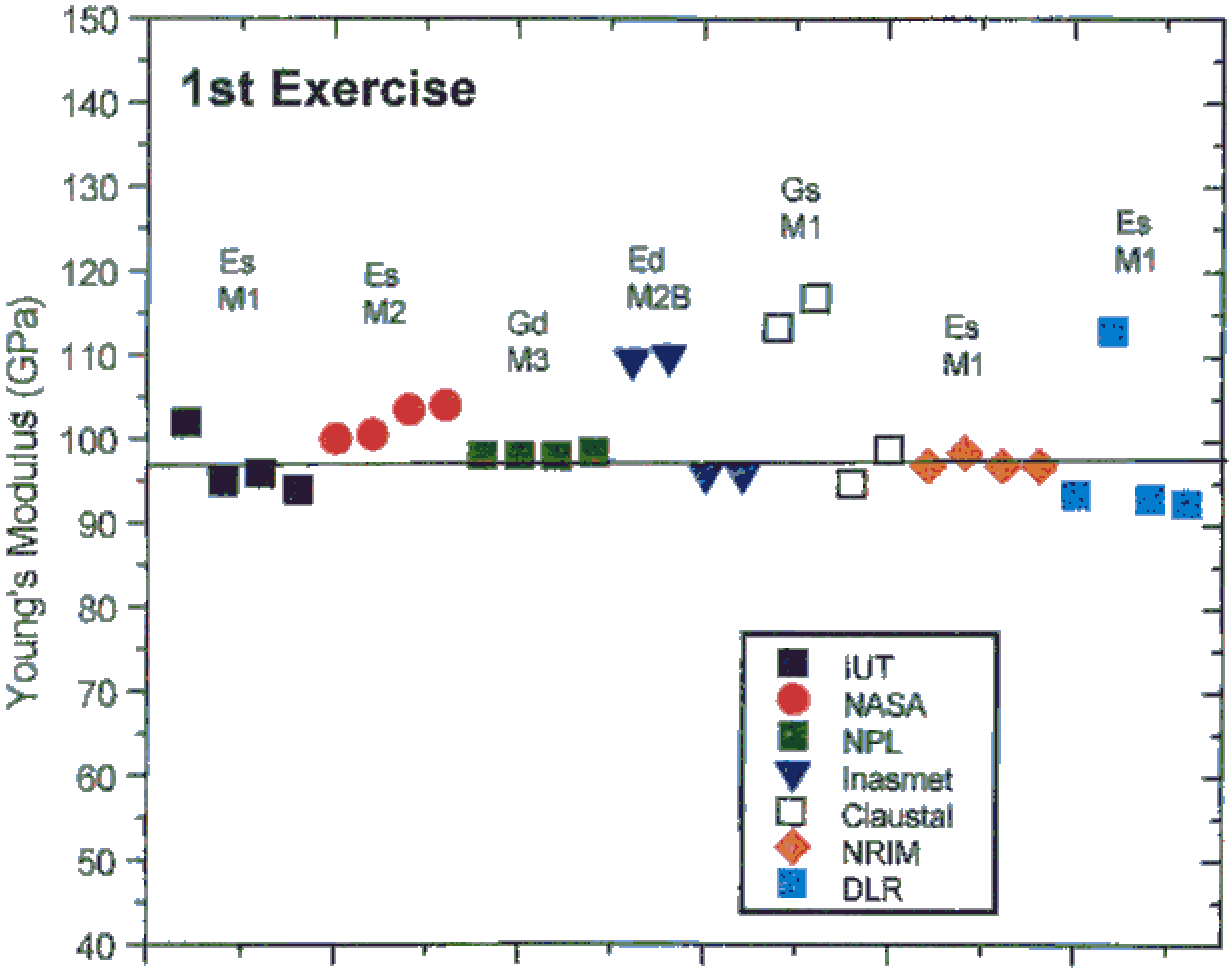

Figure 3: Variation of Young's Modulus Data at 200°C (from VAMAS Intercomparison Exercises [12,13])

Proposed Revision To MMC Tensile Test Method - for

the Elevated Temperature Measurement of Young's Modulus

For the accurate measurement of Young's modulus it is recommended that a separate test is carried out in addition to that used to measure the full stress-strain curve, following the procedure outlined in Refs 14, 15. In the procedure, the Young's modulus is calculated from the elastic part of the stress-strain curve, obtained during a tensile load-unloading test. Double-sided strain measurement is recommended and the method can be applied equally to room temperature and elevated temperature modulus measurements. Provided that no gross plastic deformation or microstructural changes occur the same specimen can be tested over a range of temperatures.

Conclusions

Results from work at NPL support the utility of a separate test procedure for the modulus measurement of MMC.

Because the reduction in modulus over the typical temperature range considered is small and MMCs generally exhibit only limited elastic behaviour, accurate strain measurement is vital for obtaining reliable modulus data. Data from the VAMAS and other intercomparison exercises confirm that double-sided strain measurement is necessary for accurate modulus measurement from the tensile test.

The dynamic modulus measurements made using the IET showed excellent repeatability, with a typical uncertainty of ±1%. Dynamic methods have the advantage of being quick and simple, but the issue regarding the relevance of dynamic vs static measurements has still not been fully addressed.

The results from tensile tests carried out using high temperature strain gauges were in good agreement with the IET data up to about 200°C, but above this there were large discrepancies in the values obtained from the two techniques. Although a proper strain gauge installation can provide the required accuracy and precision of measurement to give excellent modulus data, it does require a certain expertise and there can be considerable effort and time in preparation, and this might not always be a practical option. Further work is also required on the different strain gauge/adhesive combinations to evaluate their performance further.

Summary

- The reduction in modulus of the MMC and aluminium alloys over the temperature range considered is small, typically 6-8% on heating to 200°C, and 12-14% up to 300°C.

- Data from strain gauge tests carried out according to the proposed test procedure were in good agreement with the IET data up to 200°C.

- For tensile tests, double-sided strain gauges or extensometry should be used to take into account bending in the specimen which can seriously affect the modulus value measured. If possible, a Class 0.2 extensometer should be used.

- A new tensile test procedure has been proposed which recommends high precision strain measurement and loading within the elastic limit. The method is applicable to metals and other materials, and will be considered for submission as a separate part of ISO/TTA2 - the tensile test procedure for MMCs.

- Demand still exists for a simple, inexpensive, high resolution double-sided extensometer for accurate strain measurement.

Acknowledgement

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