MATC (MN) 33

Determining elastic moduli of hardmetals

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The measurement of elastic moduli of stiff brittle materials to high temperatures has been made using a simple method developed for ceramic materials and cited as an Annex to ASTM C1259. This method is based on impact excitation of disc test-pieces. Measurements have been made on a variety of hardmetals at room temperature, and the method has been found to work well. The dimensional accuracy requirements for the test-pieces have been determined by studying as-fired and cylindrically ground specimens. Selected hardmetals have been measured to high temperature, limited by practical considerations of oxidation in the available system. The temperature dependencies of elastic moduli are similar but not exactly the same for different grades.

Introduction

Increasingly elastic modulus measurements are required for numerical modelling purposes as input to finite element calculations, yet are often inaccurately measured or reported, especially for brittle materials where strains to failure are small, and strain-gauging techniques therefore not particularly accurate. This Measurement Note reports a study of the applicability of the impact excitation (natural frequency) method applied to disc test-pieces, which are usually readily prepared. This method is given as an annex in ASTM C1259 for advanced technical ceramics, but is not widely used or reported.

Test method

The method involves striking a disc test-piece and detecting the vibration modes. When struck at the centre, an axisymmetrical, ‘diaphragm’ mode results, with a circular node at approximately 0.68 of the disc radius. If the disc is struck on the nodal circle, the principal mode of vibration is ‘saddle-shaped’. This latter has a distinctly lower frequency. The ratio of these frequencies, coupled with the disc thickness to diameter ratio, gives Poisson’s ratio, ν, through a look-up table or algorithm. Either of the frequencies combined with the disc dimensions and mass gives Young’s modulus, E. Young’s modulus and Poisson’s ratio then gives shear modulus, G. Unlike other methods, the disc method provides Poisson’s ratio directly, rather than by combining separate measurements of E and G, which is inherently a less accurate method. The formulae are given in the standard

Practically, the disc can be struck with a ball on a plastic strip allowing a single strike to be made, the correct position being targeted. The induced vibrations can be detected using a microphone or piezoelectric sensor, and the natural vibration frequencies are determined using a Fourier transform frequency analyser, from which the relevant frequencies can be identified. In practice, the ratio of the two relevant frequencies must fall within a range of 1:1.4 to 1:1.7. From prior approximate knowledge of E and ν, the relevant frequencies can be estimated, so narrowing the search and homing in on the correct peaks. A commercial IMCE RFDA System 23 apparatus was used.

To extend the test to elevated temperature, it is necessary to replace the striker with a remote tapping device. In the present work, small ruby balls, 2 mm in diameter, were dropped down a tube to strike the test-piece surface. Striking near the edge excites both relevant modes of vibration, allowing both frequencies to be measured for a single strike. The vibration spectrum was detected using a piezo-sensor to which a long sensing rod of very low, and thus non-interfering, vibration frequency was attached to reach in through an aperture in the furnace wall and to rest on the test-piece surface near its edge. As the temperature is raised, the frequency shift is followed. For this work, a Lemmens Elektronika BV ‘Grindosonic’ Mk V and a Hewlett Packard ‘Dynamic Signal Analyser No. 35665A’ were used, previously calibrated against independent counter-timers. The test-pieces were supported on a small pad of ceramic fibre insulation titled at a small angle to the horizontal to ensure the ruby balls rolled off after contact and did not interfere with subsequent measurements

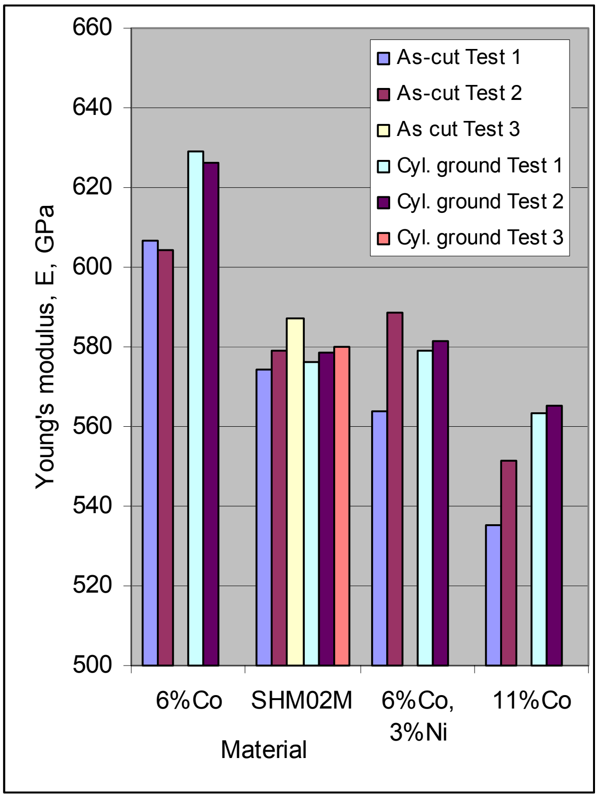
Test materials

Test materials of various grades were supplied by UK-based manufacturers in the form of discs (Table 1).

Table 1 – Materials used

Supplier	Material grade/code	Density, Mg/m^3
1	6% Co	14.96 ± 0.01
	6% Co/3% Ni	14.50 ± 0.02
	11% Co	14.36 ± 0.01
2	9%Co	14.44 ± 0.02
3	8.5% Co	14.81 ± 0.02
	10% Co	14.45 ± 0.02
	15% Co	14.02 ± 0.02

The discs were about 2 mm thick and 30-40 mm diameter prepared by grinding on both faces, initially without peripheral grinding, and later peripherally ground by mounting onto a mandrel and cylindrically grinding.



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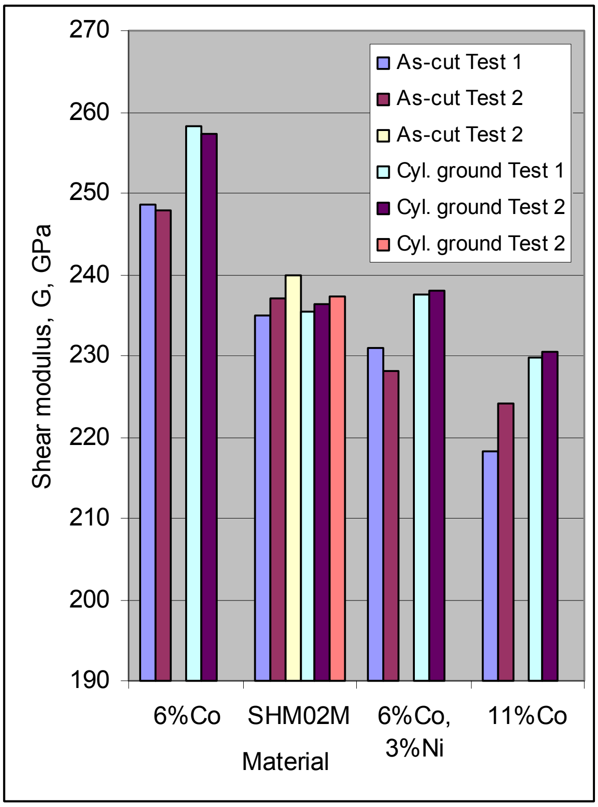


(b)

Figure 1: (a) Young's modulus, (b) shear modulus, for four materials, showing improvements in reproducibility after cylindrical grinding.

Room temperature measurements

Initial tests were made on disc samples pressed to shape or cut from bars. The as-sintered bars were not perfectly round, those from supplier 1 having a flattened region, and those from supplier 3 being oval. The result was some scatter between test-pieces, and in the case of SHM02M, the frequency peaks showed some doubling. Figure 1 shows the results for *E* and *G* obtained using average thickness and diameter of the discs. After cylindrical grinding the roundness was improved from ±0.1 mm to better than ±0.01 mm, and the reproducibility of the measurements was much improved. The two frequencies of interest for this work were in the range 10 -12 kHz and 15 -18 kHz for the sizes of test-piece used.

Figure 2 shows a correlation of the measurements of Young's and shear moduli and Possion's ratios with total nominal metal content, compared with correlations computed using Doi *et al.*'s data (2) and average data trends from Brookes (3). The Doi correlation seems to predict lower values of *E* than measured, while the correlation with Brookes' data seems nearer to actuality. It should be noted that in all cases porosity has been ignored, although small amounts may have a secondary effect on the results.

High-temperature measurements

Tests were made on discs from supplier 1 after cylindrical grinding. The temperature was raised in a series of steps and allowed to equilibrate for 10-15 minutes before making the measurements. It proved readily possible to follow the shift in frequency with temperature up to typically 700 °C, but oxidation in the unsealed furnace system, despite argon gas flushing, limited the value of the results. Consequently test temperatures were limited to 500-600 °C at which mass oxidative gains were negligible.

Figure 3 shows the results for the 6% Co hardmetal. There is a clear non-linear drop with increasing temperature for both *E* and *G*, while Poisson's ratio increases slightly with

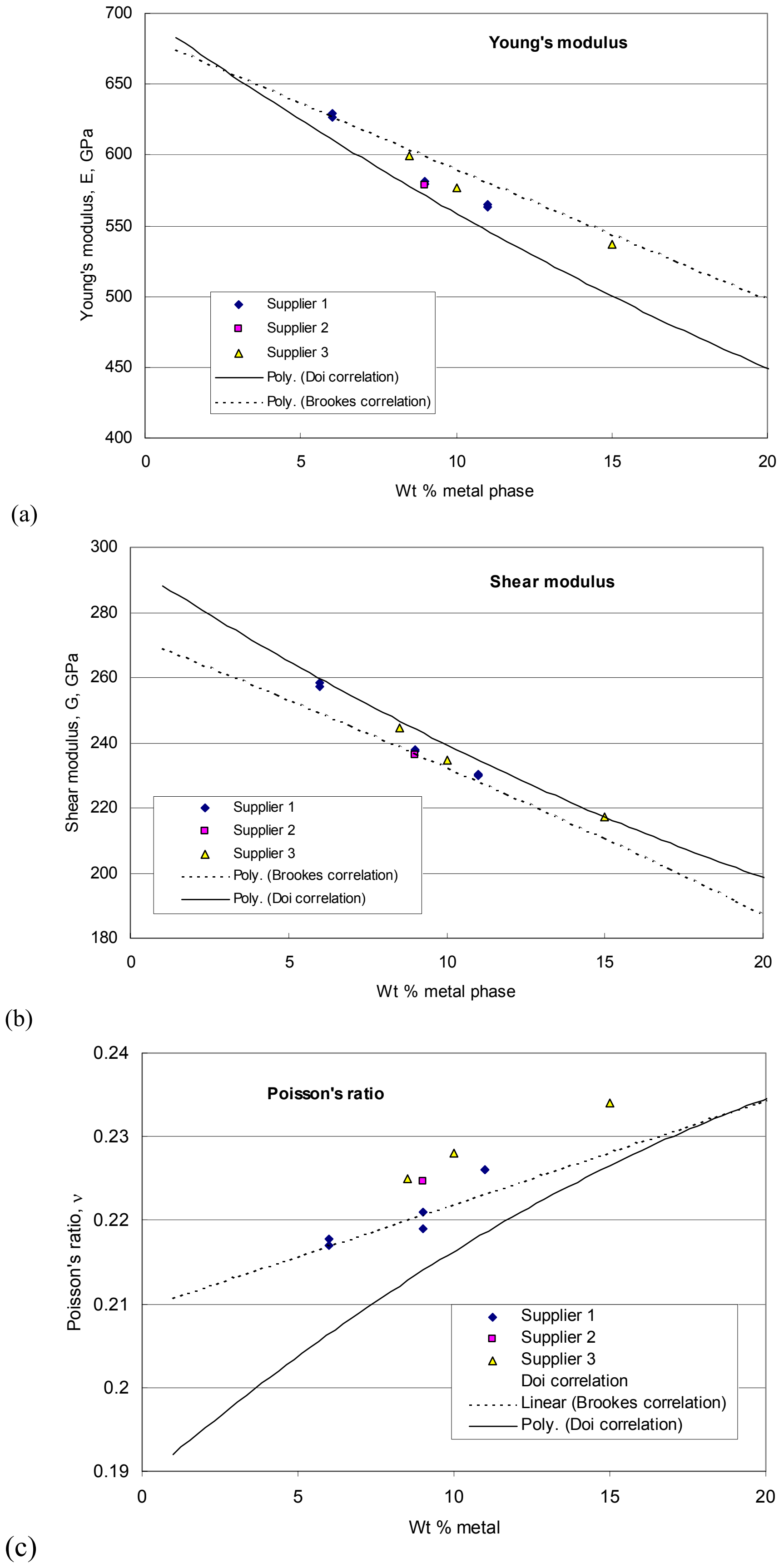
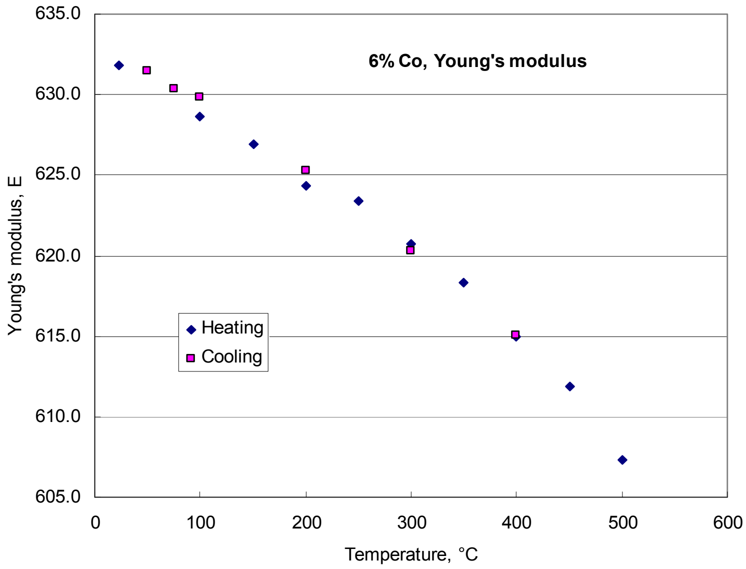


Figure 2: Correlation of measured (a) Young's modulus and (b) shear modulus with total metal content, compared with correlations calculated from the data of Doi *et al.* (2) using research data and by Brookes (3) using commercial data.



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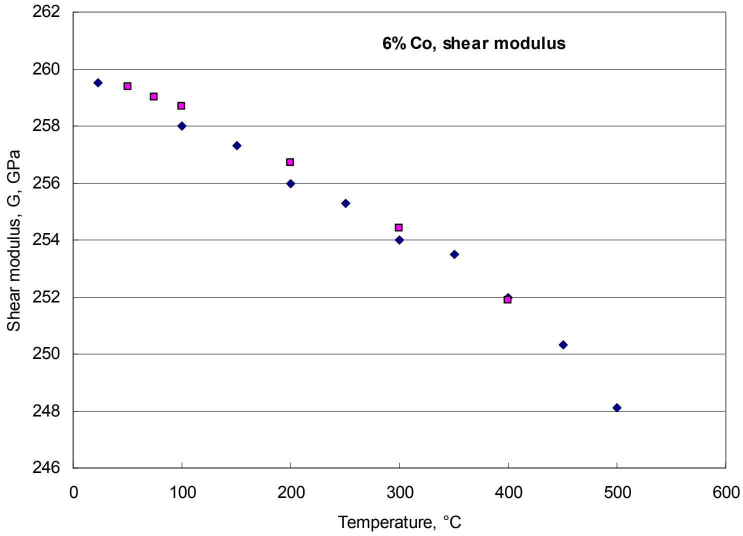

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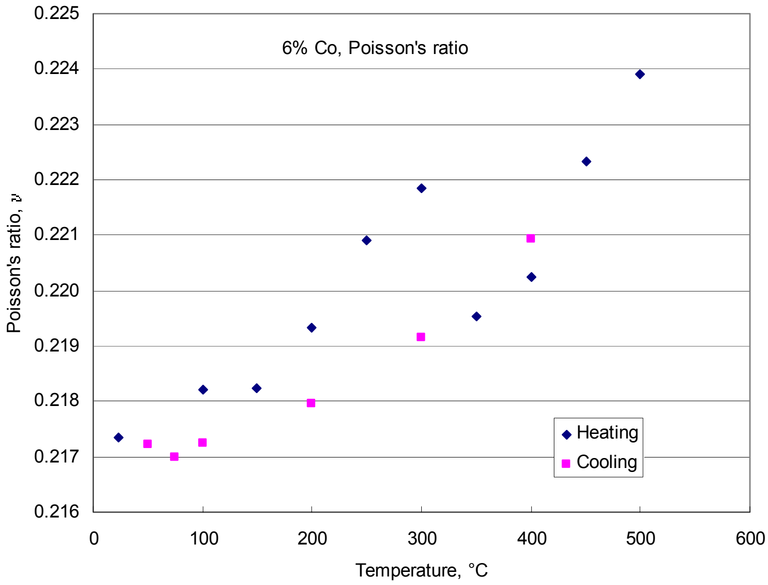


(c)

Figure 3 – (a) Young's modulus, (b) shear modulus, and (c) Poisson's ratio as a function of temperature for 6% Co hardmetal

increasing temperature. The scatter in the data probably originates from variable deviations between the furnace thermocouple temperature and the actual test-piece temperature in the presence of argon gas flushing.

Tests on the 6%Co/3%Ni and the 11% Co materials showed similar trends, although the former of these showed some unusual fluctuations in data during the first thermal cycle which may be related to annealing effects in the mixed binder phase. In order to establish whether there is a universal relationship for the temperature dependence, the data were normalised using the room temperature value, and the plots are shown in Figure 4. There is a small variation in the trends with temperature, suggesting that the materials behave similarly but not equivalently. The approximate linear temperature coefficients up to about 300 °C are:

E: -48×10^{-6} °C^{-1},
G: -54×10^{-6} °C^{-1},
ν: $+44 \times 10^{-6}$ °C^{-1}.

These data are all uncorrected for thermal expansion of the materials, i.e. are based on room-temperature dimensions.

Oxidation seems to be clear limitation to the test in the present facility. The natural frequencies could probably be measured to temperatures higher than 700 °C without difficulty if an inert atmosphere can be maintained. This would need modifications to the equipment to have a sealed furnace, replacing the ball-drop mechanism with a striker as used by the IMGC system, and detecting the frequencies using a microphone and waveguide, rather than a piezo-transducer.

Error analysis

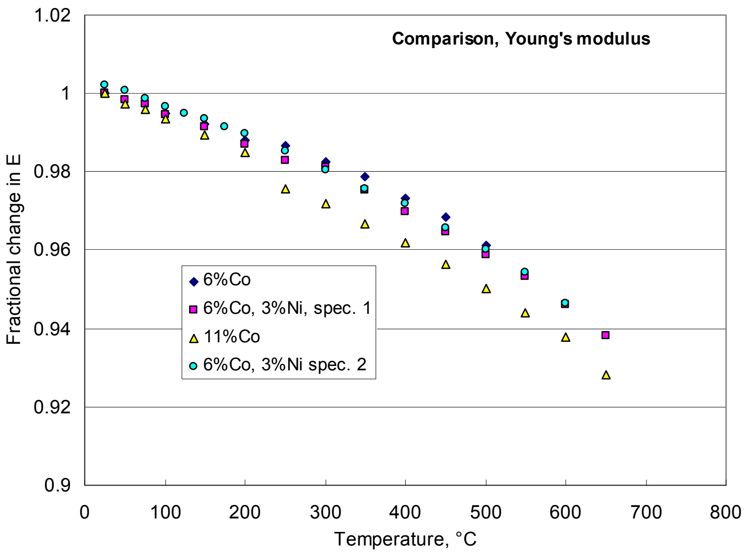
For the typical dimensions and mass of the discs used in this work, the Table 2 shows the contributions of each practically achievable measurement accuracy level to the accuracy of the results. It can be seen that the principal source of error is the thickness, so it is important to achieve good parallelism and to measure to the nearest 0.002 mm. The second most important factor is the diameter, and as this work has shown it is desirable to cylindrically grind as-sintered discs or slices from bars to improve roundness and reduce errors. In contrast, mass and frequency measurement provide minor contributions. Allowing for both measurement accuracy and dimensional perfection, an overall accuracy of better than 1% in Young's modulus should be readily attainable. The accuracy of the shear modulus is slightly worse. Poisson's ratio can be determined only to an accuracy of about 5% overall, and this accounts for the greater scatter seen in Figures 3(c) and 4(c).

Table 2 – Error analysis

Parameter	Practical accuracy	Effect on elastic property		
		E	G	ν
Diameter	0.01 mm or 0.03%	0.07%	0.04%	1.3%
Thickness	0.002 mm or 0.1%	0.12%	0.30%	1.1%
Mass	0.0005 g or 0.002%	0.03%	0.04%	0.3%
Freq. 1	0.005 kHz or 0.03%	0.02%	>0.04%	1.1%
Freq. 2	0.005 kHz or 0.05%	0.05%	0.04%	1.6%
Additively combined		0.3%	0.46%	5.2%

Summary

- The disc impact excitation method has been found to be a quick and simple way of measuring elastic properties of hard-metals at room and elevated temperature.
- It can produce accurate data provided that the test-pieces are of accurate dimensions and of homogeneous density. To make measurements accurate to about 1% for *E* and *G*, the necessary actual dimensional precision (and measurement accuracy) on test-pieces are considered to be:
  Parallelism of faces: ±0.002 mm
  Roundness of perimeter: ±0.01 mm
- The example data produced are in line with extant correlations with wt% metal phase.
- The high-temperature variation of elastic properties can be determined by this technique, limited by the ability to restrict oxidation. Similar but not equivalent trends are shown by materials of different binder phase content.



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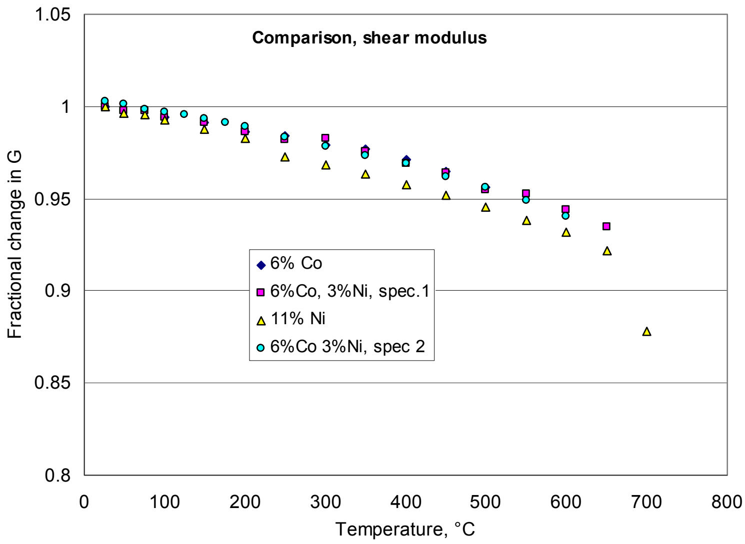

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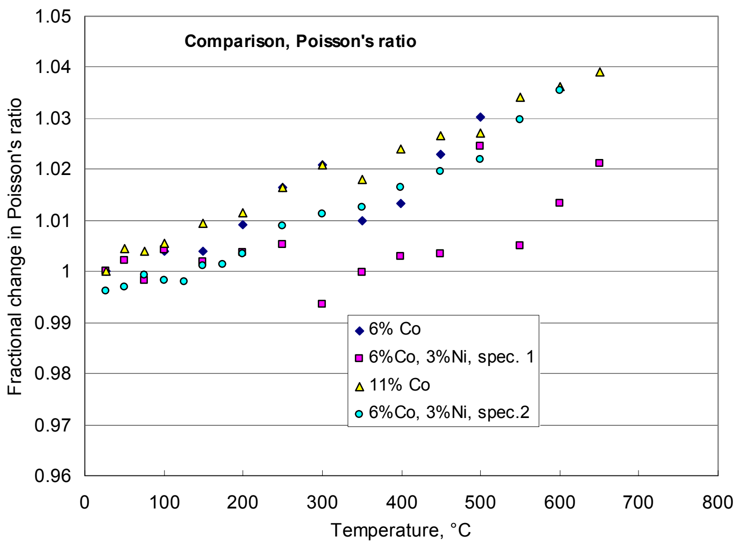

(c)

Figure 4 – Fractional changes in elastic properties as a function of temperature. The changes appear to be larger for the 11% Co material than for the others.

Acknowledgements
This work was conducted as part of the DTI Materials Metrology Programme at NPL.

References
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(2) Doi, H., Fujiwara, Y., Miyake, K., Oosawa, Y., A systematic investigation of elastic moduli of WC-Co Alloys, *Met. Trans. A*, 1970, **1**(5), 1417-25.
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