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Evaluating the mechanical properties of metallic glass wires by nano-indentation

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Materials

ABSTRACT

Purpose: Metallic glass wire products are known for their high fatigue strength and their interesting magnetic properties. The thermo-kinetic requirements for fully amorphous structure in these cast alloys require rapid solidifications of the order of 10^6K per second. This condition imposes a section thickness of less than 200μm in diameter for fully amorphous structured wires. This unusual dimension makes it difficult to use the traditional pull type tensometric devices for characterising the mechanical properties of these high strength wires. The advent of nano-indentation techniques provide a new way of easily determining the mechanical properties of these high strength thin sectioned wires. This work reports the use of nano–indentation system to characterise the mechanical properties of Fe(Cr)SiB metallic glass wires
Design/methodology/approach: The analysis of the averaged load displacement data from nano-indentation experiments allowed the use Hertzian indentation mechanics for the elastic and plastic contact and from which a representative stress-strain curve can be determined.
Findings: The elastic modulus determined for the wire varied from 150–160GPa after corrections for the effect of indenter. The hardness and representative strength were respectively of the order of 1000Hv and 4000MPa.
Research limitations/implications: These mechanical indices (except modulus) compared favourably well with measurements from other combinations of established techniques. The elastic modulus values were slightly lower than those reportedly determined by acoustic methods.
Originality/value: This work emphasised the value of nano-indentation method as a relatively non destructive method for the mechanical characterisation of thin sectioned high strength fibres.
Keywords: Metallic alloys; Amorphous materials; Mechanical properties

1. Introduction

1.1. Background to metallic glass formation

While amorphous structure is a familiar occurrence in non metallic melts, it is only relatively recent in history that the first amorphous metals were prepared[1]. The solidification of metallic melt into non crystalline amorphous structure is considered to be the extreme state of solid metastability.

Process-wise, the vitrification of metallic melt is an extremely difficult process because of the highly favourable thermo–kinetic factors for the orderly arrangement of atoms on solidification. Many reviews now exist on the subject of metallic glass formation (see eg, ref[2]), and the details accompanying the vitrification of metallic melts are now well established. Generally, the transformation of a melt from liquid to solid could be depicted on a temperature–time–transformation (T–T–T) diagram as in Figure 1. Theoretical calculations [2] from such T–T –T plots suggest that critical cooling rates CCR for glass formation for pure metals are higher than 10^{13}K/s. Hence reports

of amorphous pure metals are limited to films prepared from methods like vapour depositions and ion implantations that involve atom by atom build up. Alloying has been used to considerably lower CCR. A host of readily glass forming (RGF) alloys with CCR within the range achievable in rapid solidification (rs) processes are now available. A detailed listings of RGF alloy families is provided in ref[3]. Commonly available RGF alloys (which includes the Fe-Si-B family), would normally consist of metalloid (B & Si) contents of about 10 to 30 atomic percent [4–6].

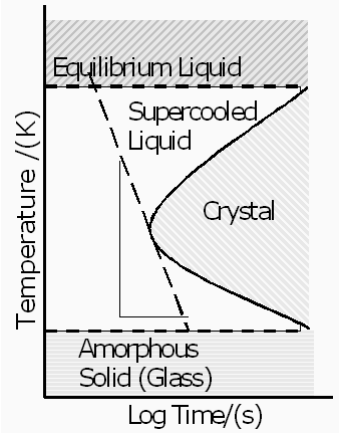

Fig. 1. Temperature time transformation diagram for a liquid.-solid transformation

Cooling rates of the order of 10^6K/s in rapid solidification translate to section thickness of about of 10 to 60μm thickness of ribbon [7,9] or 100 to 200μm diameter of wire[10,11]. These rapidly quenched products are unique for their engineering properties and a number of potential applications have been suggested in structural, electronic and magnetic devices. The typical section diameter of about 100μm of a high strength wire shaped material in itself presents a different challenge of handling for mechanical characterisation. A number of methods have been used in literature and in this work a new approach using nano-indentation method is being highlighted.

1.2. Methods for mechanical characterisation of metallic glass wire

Because of the unconventionally thin section combined with very high strength, the mechanical evaluation of metallic glass wires require specialised and sometimes unconventional procedures. Static strength has been most widely reported as microhardness and fracture stress [10]. Microhardness is more commonly used as it appears to be the simplest and even in some cases the only sensible technique. Most commonly, Vickers hardness using a pyramidal diamond indentor has been used with varying loads. This technique though provided some measure of mechanical properties, is highly susceptible to errors especially at low loads when hardness is independent of loads since indentation is very small. On the other hand, higher loads are precluded since the resulting indentations become too large in relation to sample dimensions. Other alternative of the use of knoop indentor, which gives added advantage of a biased diagonal in the longitudinal section, allowed the use of larger loads. Overall such microhardness values could only give hardness numbers and cannot provide direct information on the yield strength and other elastic and plastic properties for full mechanical evaluation. Traditional pull tests could only at best provide information on fracture stress and it is difficult to get information on elastic properties and elastic-plastic transition because gripping such a high strength fibre in any of the known standard methods could not avoid slipping or localised damage at the gripped ends.

1.3. Nano-indentation

The advent of nano indentation techniques provides new tools for mechanical characterisation of materials on a micro and nano scale. These enable indentation with very small force typically of the order of μN to mN. Instrumentation has been developed to allow the acquisition of the force versus indentation depth data. Analyses of such data in the loading and unloading cycle allow the separation of indentation data due to elastic deformation. Since resolution of depth measurement is in the order of 1nm, it is possible to analyse the response of a material when subjected to extremely low loads over a small area typically by a microscopically tipped indenter. The mechanics of deformation for spherically tipped indenter have been proposed[12 – 14] and are now well established. It is now possible to generate representative indentation stress–strain data based on the primary load – depth data and the characterisation of the geometry of the indenter. The elastic portion of the representative stress – strain curve, should provide a good estimate of the Young modulus, but strain at yield, and the yield strength are still the subject of many interpretations, since the mechanics of deformation under indentation though analogous to uniaxial loading is not exactly equivalent. The evaluation of theses mechanical indices depend on the application of correction factors which will vary from ductile to brittle material.

2. Experimental

Wires were directly cast from metallic melt using rotating water bath process as described earlier[11]. The amorphous structures of the wire were confirmed using X-ray diffraction (XRD) and differential (DSC). Typically wires were fully amorphous as characterised for diameters in the range 80 – 100μm. For indentation, wires were mounted in a cold setting resin and polished to 1μm finish. The indentation experiments were performed using UMIS 2000; a force driven micromechanical probe. A spherically tipped indentor of radius 1μm was used. The true equivalent radius was corrected for at every depth by generating a radius file to apply the correction for minor geometrical discrepancies. The indentations were made on a line at 20μm spacing and for every experiment a total of 20 sets of data were averaged to generate and average force–depth data. For each indentation, penetration was measured over 50 load steps using multiple load–partial unload sequence.

3. Results and discussion

A typical force penetration data obtained for FiSiB wire is shown in Figure 2. The unload data points represents completely elastic response, while the load data points represents the elastic–plastic response. The two sets of data points are coincident for completely elastic response and the elastic plastic transition can be seen when variation exists between the load and unload data point as indicated by the arrow in Figure 2.

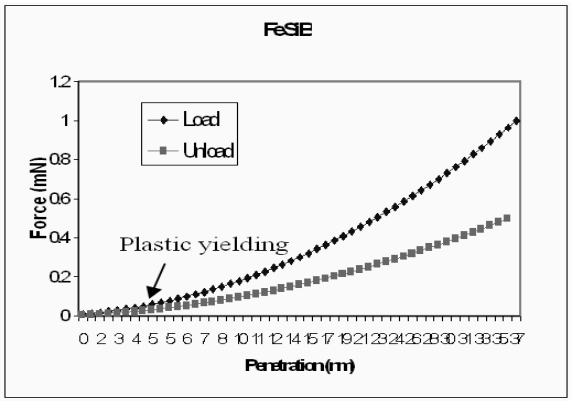

Fig. 2. Force depth relationship from nanoindentation for FeSiB wire

This data was processed further to produce a representative stress – strain data. A typical graph of representative stress strain is shown in Figure 3 for Fe CrSi B wire. The yield can be detected as in a conventional pull-type tensile test. A composite indentor–material modulus E^* could be obtained at every depth utilising only elastic information. Figure 4 shows the variation of E* for the two types of wire compositions used. Ideally E^* should be constant since the material is isotropic. Though this results showed an initial instability in the values of E^*, for both cases, these settled to almost constant values around 200Gpa. The true value of Material modulus E_m, can be separated from that of the indentor (E_i) according to the equation[5,6]:

$$\frac{1}{E^*} = \frac{1-v_m^2}{E_m} + \frac{1-v_i^2}{E_i}$$

From the knowledge of indentor properties (E_i and v_i), the true modulus E_m of the material is computed. Values obtained from literature for diamond were used for the indentor to obtain final values of material modulus.The overall mechanical properties for the two compositions of MG wires are given in Table 1. Values of similar properties obtained by other methods are given in Table 2.

The values of hardness for both of amorphous alloys that resulted from microhardness test and UMIS test are around 900 – 1100 Hv. These values are broadly consistent with other published data [10, 11, 15] for metallic glass wires of similar compositions. The values of modulus of elasticity are around 130 – 160 GPa. These modulus values are slightly lower than expected from published data but are generally of the same order of magnitude. These variations may be due to the testing conditions, in the micro-mechanical test, The deformation here is of the order of nanometers, and slight movement of the sample on the resin, would upset values considerable. Additionally the primary output from the data acquired is a composite value E^*. The accuracy of E_m depends on the use of true values for indentor properties v_i and E_i for which at the moment values for pure diamond were used while indeed the indentor is a diamond coated tip. Nonetheless, the fact that the determined modulus values were less than 10 % out from measurements obtained from a relatively non-destructive testing is a significance advance in the micro-mechanical testing of amorphous wire. From the indentation test results, a representative stress and strain graph can also be obtained. The strength and the strain of the samples and comparing with the result from tensile test are given in Tables 1 and 2.

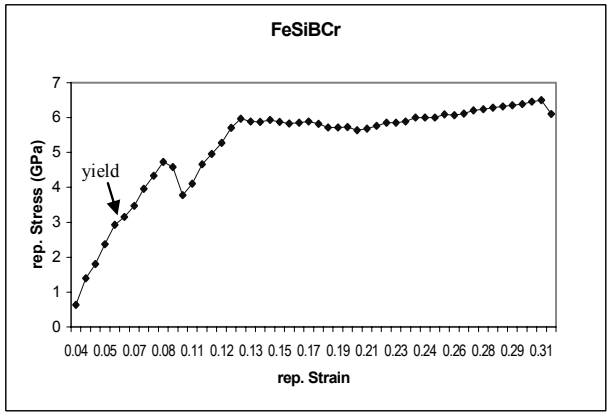

Fig. 3. Representative stress – strain relationship obtained for FeCrSiB metallic glass wire

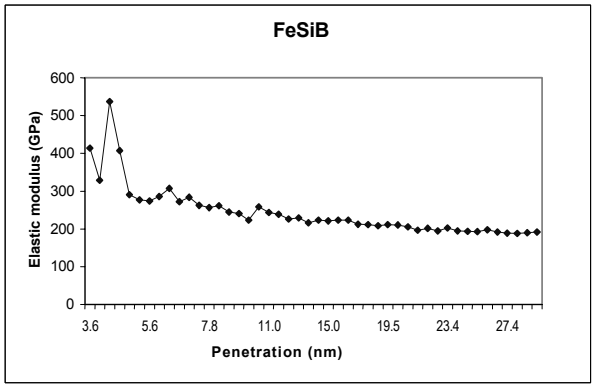

Fig. 4. Variation of composite modulus with depth

Table 1.
Static mechanical properties of amorphous alloy wire samples resulted from nanoindentation test

Alloy	Yield(σ_y) MPa	Yield strain(ε_y)	Modulus (E) GPa
Fe-Si-B	3390	0.085	146.922
Fe-Si-B-Cr	3960	0.070	146.524

Table 2.
Static mechanical properties determined from uniaxial

Alloy	Fracture strength (MPa)	Fracture strain (%)
Fe-Si-B	3180	2.6
Fe-Si-B-Cr	3550	1.7

The correlation between strength/hardness and modulus of elasticity for a variety of amorphous alloys had been investigated and generally for amorphous alloys the ratio is found to be:

$$\sigma_y/E = C_0$$

The ratio (C_0) (i.e., plastic strain) is almost constant, it lies between 1/60 (0.0166) and 1/40 (0.025) with several observations. It indicates that the basic flow mechanism might be similar in all amorphous structures at least in each system. The present measurements indicated the strength–modulus (σ/E) ratio for the glassy wires to be ~ 1/40 compared with σ/E of the order of 1/100 normally associated with high strength crystalline metals. The higher σ/E ratio observed for amorphous alloys are generally attributed to the absence of crystalline defects.

4. Conclusions

Nanoindentation offers a convenient way of evaluating the mechanical properties of sub millimetre diameter high strength metallic glass wires. The multiple load – partial unload sequence was used to generate force indentation depth data that allowed the determination of the elastic and yield properties. The modulus of elasticity estimates from this work is slightly lower than values obtained from other techniques, because there is still need to allow for corrections for slippage of wire samples and the elastic strain recorded might be slightly out. The static strength however is consistent with previous microhardness and fracture strength form classical tensometer tests.

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