### Short communication

# Refinement and characterisation of a thermodynamically stable $\beta$ -[Ni(AI,Ti)]- $\beta$ '-[Ni<sub>2</sub>AITi]- $\gamma$ '-[Ni<sub>3</sub>(AI,Ti)] metal-metal composite

J. D. Robson and H. K. D. H. Bhadeshia

The Ni-Al-Ti system contains three intermetallic phases,  $\gamma'$ ,  $\beta$ , and  $\beta'$  which have complementary properties. The objective of the present investigation was to coextrude these three phases to form a metal-metal composite (MeMeC) and investigate the mechanical properties and failure mechanisms of such a material. High strengths, comparable with that of the strongest constituent phase, have been recorded for the MeMeC material. Strength is maintained at a constant level up to 500°C above which it falls progressively. The compressive strain to failure is considerably greater that that expected for monolithic  $\beta$  or  $\beta'$  phases. It has been shown that the interfaces between nominally single phase regions provide lines of weakness along which cracks propagate during compressive loading. Preliminary attempts to strengthen the interfaces by a 24 h heat treatment at 900°C have been carried out.

At the time the work was carried out the authors were in the Department of Materials Science and Metallurgy, University of Cambridge, Pembroke Street, Cambridge CB2 3QZ, UK. Dr Robson is now in the Manchester Materials Science Centre, Grosvenor Street, Manchester M1 7HS, UK. Manuscript received 6 September 1999, accepted 14 October 1999. © 2000 IoM Communications Ltd.

#### Introduction

The intermetallic compounds which are under consideration for use in aerospace applications often suffer from either poor ambient temperature ductility or insufficient elevated temperature strength. One promising route for obtaining the desired properties is to combine various intermetallic phases with complementary properties. Several workers have achieved finely dispersed multiphase intermetallic materials by directional solidification of ternary or quaternary alloys.<sup>1-4</sup> A different production route was used by Hsuing and Bhadeshia<sup>5</sup> to create a multiphase Ni-Al-Ti alloy. Three single phase powders were coextruded to give a three phase  $\gamma' + \beta + \beta'$  metal-metal composite (MeMeC). The aim of the present work was to refine the structure of such a material and investigate in more detail its mechanical properties. In particular, the ways in which the mechanical properties of the MeMeC material vary with temperature are reported here for the first time.

#### **Experimental and observations**

Three powders were manufactured, each designed to consist of one of the three constituent phases (see Table 1). Following Hsuing and Bhadeshia, the composition of each powder was chosen to correspond to the appropriate corner of the equilibrium tie triangle containing the three phases. The  $\beta$  and  $\beta'$  powders were prepared by mechanically crushing cast ingots which proved more economical and less problematic than the gas atomisation route used by Hsuing and Bhadeshia. Because  $\gamma'$  has a higher ductility and is not easily pulverised in this way the  $\gamma'$  powder was, therefore, produced using argon gas atomisation. The mean size of the  $\beta$  and  $\beta'$  powder particles was 50 µm and the mean size of the  $\gamma'$  particles was 100 µm. The powders used here are considerably finer than those used by Hsuing and Bhadeshia which had a minimum size of 150 µm.

The powders were analysed using X-ray diffraction which revealed that, in each case, small quantities of secondary phases were also present (e.g.  $\gamma'$  was present in both the  $\beta$  and  $\beta'$  powders).

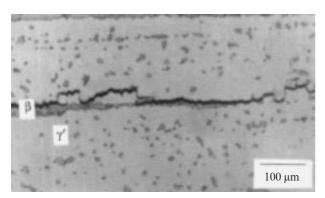
In an earlier work,5 two variants, MeMeC B  $(50\gamma'-25\beta-25\beta')$ , wt-%) and MeMeC C  $(70\gamma'-15\beta-15\beta')$ , wt-%) were manufactured. Preliminary tests showed that in both materials some of the strength could be usefully sacrificed to obtain increased ductility. In an attempt to realise this, the quantity of the more ductile  $\gamma'$  phase was increased to 80 wt-\%, with the  $\beta$  and  $\beta'$  fractions reduced to 10 wt-% each. For consistency with the previous work this variant is identified as MeMeC D. The three powders were therefore mixed in these proportions and coextruded. A set of trials was conducted which found the optimum extrusion conditions to be a preheat to 1170°C for 70 min followed by extrusion with a reduction ratio of 16:1 at a ram speed of  $25 \text{ mm s}^{-1}$  using double E glass and fibre glass lubricant. The microstructure of as extruded MeMeC D is shown in Fig. 1 and it can be seen that the desired intimate dispersion of the three constituent phases has been achieved. Detailed microscopy revealed the presence of secondary phases in the nominally single phase regions. In particular  $\beta'$  precipitates were observed in the  $\gamma'$  regions. Heat treatments were performed in the temperature range 700-1000°C for times up to 1000 h followed by microscopy. The results were consistent with the previous work<sup>5</sup> confirming the as extruded structure has a high degree of stability.

Compression tests were performed using a 50 kN servohydraulic testing machine on  $2.5 \times 2.5 \times 6.0$  mm rectangular

Table 1 Compositions, given in at.-%, of three corners of 900°C  $\gamma'$ - $\beta$ - $\beta'$  tie triangle:<sup>6</sup> actual compositions of three alloy powders are given in parentheses

Phase	Ni	Al	Ti	_
γ'	72·7 (70·8)	12·9 (13·5)	14·4 (15·7)	
β	56·5 (56·1)	35·9 (36·3)	7·6 (7·6)	
β'	53·2 (52·7)	25·1 (25·8)	21·7 (21·5)	

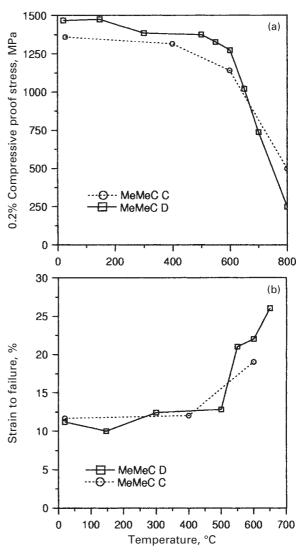
1 Microstructure of as extruded MeMeC D (optical)



2 Micrograph showing crack observed after interrupted room temperature compression test (optical): compression axis is horizontal

blocks cut from the extruded bar in either a longitudinal or transverse orientation. Interrupted tests were also performed where specimens were compressed to 90% of the failure strain and then examined. In these specimens cracks were observed, the majority of which lay along the interfaces between the nominally single phase regions. In an attempt to strengthen the interfaces several specimens were heated to 900°C for 24 h. It was found that this pretreatment did not make any significant difference to the results obtained in compression tests. An optical micrograph showing a typical crack in a specimen from an interrupted test is given in Fig. 2 (this specimen had been heat treated prior to testing). The crack has propagated along an interface between the  $\beta$  and  $\gamma'$  phases. It is interesting to note that the crack leaves the interface at one point and instead seems to take a path following a line of closely spaced  $\beta'$  precipitates in the  $\gamma'$  matrix. When this line stops the crack reverts to propagation along the interface. In this example the crack can be seen to reach the end of the  $\beta$ region and can then only continue by passing through the  $\gamma'$ . When this happens it can be seen that the crack path becomes much more tortuous, jumping between  $\beta'$  precipitates. These observations indicate that the presence of brittle  $\beta'$  precipitates in the  $\gamma'$  matrix may be detrimental to toughness by providing an easier crack path through this phase.

Elevated temperature compression tests were then performed at temperatures up to  $800^{\circ}\text{C}$  on specimens of both MeMeC C and MeMeC D. The 0.2% compressive proof stress and compressive plastic strain at failure are shown for MeMeC C (70%  $\gamma'$ ) and MeMeC D (80%  $\gamma'$ ) in Fig. 3. It can be seen that a constant proof stress is maintained up to  $\sim 500^{\circ}\text{C}$ , above which the strength falls. The fall in proof stress above  $500^{\circ}\text{C}$  is accompanied by a significant progressive increase in the plastic strain to failure. Above



3 Variation with temperature of a 0.2% compressive proof stress and b mean plastic strain to failure for composites MeMeC C and MeMeC D

650°C failure had not occurred after 30% strain, by which time the specimen had barrelled significantly. It is also interesting to note that despite the high  $\gamma'$  content of MeMeC D (80 wt-%) the characteristic peak in strength of  $\gamma'$  is not detected in the MeMeC material. Comparing the two variants, little difference is observed between the strains to failure and 0·2% compressive proof stress in MeMeC C and D despite microstructural and constitutional differences. The proof stress of MeMeC D appears to fall more rapidly than that of MeMeC C between 600 and 800°C. This may be a result of the higher level of  $\gamma'$  in MeMeC D, since above the peak in  $\gamma'$  strength (at around 700°C)<sup>7</sup> the strength of this phase falls more rapidly than that of  $\beta$  and  $\beta'$ , at least until  $\sim$ 950°C (Refs. 8 and 9).

#### Summary

In summary, it has been demonstrated that a three phase intermetallic MeMeC material has the potential to offer a combination of high strength along with considerably greater compressive failure strain than monolithic  $\beta$  or  $\beta'$  intermetallics.<sup>8,9</sup> This high strength is maintained at an approximately constant level up to around 600°C, above which temperature the strength falls, more sharply in the MeMeC D variant which has a greater fraction of  $\gamma'$ .

Studies have shown that the interfaces between nominally single phase regions provide lines of weakness along which cracks propagate during compressive loading. This is in contrast to the strong interfaces observed in a directionally solidified  $\beta + (\gamma + \gamma')$  alloy<sup>1</sup> which also has a well defined Kurdjumov-Sachs orientation relationship between the brittle and ductile phases. The strong interfaces and well defined orientation relationship were identified as important in facilitating slip transfer and enhancing ductility in this alloy. Strengthening the interfaces in the MeMeC material and promoting slip transfer might therefore be expected to improve its mechanical properties. Preliminary attempts to strengthen the interfaces by a 24 h heat treatment at 900°C have been unsuccessful. This suggests that either a more severe heat treatment is required or another strategy must be employed.

#### References

- 1. A. MISRA and R. GIBALA: Metall. Mater. Trans. A, 1997, 28A, 795.
- J. D. WHITTENBERGER, R. D. NOEBE, D. R. JOHNSON, and B. F. OLIVER: Intermetallics, 1997, 5, 173.
- 3. A. MISRA, Z. L. WU, M. T. KUSH, and R. GIBALA: *Philos. Mag. A*, 1998, **78**, 533.
- 4. D. R. JOHNSON, B. F. OLIVER, R. D. NOEBE, and J. D. WHITTENBERGER: *Intermetiallics*, 1995, **3**, 493.
- L. C. HSUING and H. K. D. H. BHADESHIA: Metall. Mater. Trans. A, 1995, 26A, 1895.
- R. YANG, N. SAUNDERS, J. A. LEAKE, and R. W. CAHN: Acta Metall., 1992, 40, 1553.
- 7. N. S. STOLOFF: Int. Mater. Rev., 1989, 34, 153.
- M. YAMAGUCHI, Y. UMAKOSHI, and T. YAMANE: Philos. Mag. A, 1984. 50A, 205.
- 9. R. T. PASCOE and C. W. A. NEWEY: Met. Sci., 1968, 2, 138.

**BRITISH CERAMIC PROCEEDINGS 59** 

# ENGINEERING WITH CERAMICS

## Edited by

## W. E. Lee and B. Derby

Contents: Recollections of Roger Davidge; Foreword; Characterization of Sub-Critical Crack Growth in Ceramics Using Indentation Cracks; Freeform Fabrication of Ceramics; Biaxial Disc Flexure - Modulus and Strength Testing; Hertzian Testing of Ceramics; The Flexure Strength of Multilayer Ceramic Capacitors; Indentation Damage Zone in Calcium and Lead Titanate Ceramics; In Situ Measurements of Residual Stresses in a Thermal Barrier Coating During Thermal Cycling; Towards More Predictable Deformation in Sanitaryware Products; Influence of Environment on Delayed Failure of Alumina Ceramics; An Experimental Study of the Fracture Behaviour of Magnesium Zinc Ferrites; Erosive Wear of Alumina/SiC Nanocomposite Ceramics; Sialon/TiB2 Ceramic Composites: Synthesis, Properties and Tribology; Microstructural Requirements for Alumina/SiC Nanocomposites; Microstructure Characterisation and Superplasticity of an Alumina/Carbon Nanocomposite; The Evaluation of Novel Interphases for Oxide-Oxide Ceramic Matrix Composites; Multi-Phase Piezoelectric Structures Through Co-Extrusion; Optimisation of Ball Valve Processing Parameters; Influence of Additives to Ag Based Electrode Pastes on Bonding to PZT; The Fracture Behaviour of Dual Phase Composite Refractories; Dissolution of Dolomite and Doloma in a Model BOF Slag; Reactions of Silicon and Carbon in Reducing Atmospheres; Barium Hexaaluminate Refractories; Author Index; Subject Index

### Book 713 ISBN 1 86125 087 8 Hbk European Union £75/Members £60 Non-European Union \$150/Members \$120 p&p European Union £5.00/Non-EU \$10.00 per order

Orders to: IOM Communications Ltd, Shelton House, Stoke Road, Shelton, Stoke-on-Trent, ST4 2DR Tel: +44 (0) 1782 202 116 Fax: +44 (0) 1782 202 421 Email: Orders@materials.org.uk Internet: www.materials.org.uk



**IOM** Communications

Reg. Charity No. 1059475 VAT Registration No. GB 649 1646 11 IOM Communications Ltd is a wholly-owned subsidiary of the Institute of Materials