Tensile properties of directionally solidified AI-AI₃Ni composites with off-eutectic compositions

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Ingots ($\frac{1}{8}$ in. diameter) of Al–Al₃Ni with off-eutectic compositions were directionally solidified at two growth rates. At 1 cm h⁻¹ fibres exhibited a blade-like morphology, and at 11 cm h⁻¹, a rod-like morphology. Speciments were mechanically evaluated in tension. The composite modulus at stresses above the yield strength of the aluminium matrix obeyed the rule of mixtures, assuming ideal plastic behaviour of the matrix. An extrapolation of these data for composites with rod fibres gave a value of 146 GN m⁻² for the modulus of Al₃Ni. Tensile strength of composites with rod-like fibres followed the rule of mixtures, whereas those with non-uniform blade fibres showed a lower strength. In composites with blade-like fibres the extrapolated aluminium matrix strength was 86.3 MN m⁻², a high value attributed to dispersion hardening, and that of the fibres was 2.69 GN m⁻². Composites with blade-like fibres failed at lower strains than did those with more uniform rod-like fibres.

1. Introduction

The applicability of the rule of mixtures for estimating the mechanical properties of in situ grown fibrous composites has not yet been clearly established. Basically this rule relates a composite property, such as modulus or ultimate tensile strength to the properties of the individual component phases, weighted by their respective volume fractions [1]. The first attempt to apply this rule to a eutectic involved the Al-Al₃Ni system, directionally solidified; measured strengths were lower than those predicted by the rule [2]. Barclay et al. [3] used a rapid growth technique [4] to produce Al-Al₃Ni composites with different volume fractions of Al₃Ni fibres and measured strengths which were much lower than those predicted by the rule, presumably because of cellular microstructures generated by a non-planar solid-liquid interface. More recent work on the same system [5] has shown some agreement with the rule of mixtures, however, since the ultimate tensile strength for this eutectic has been shown to be dependent on growth rate [2, 6], additional work appeared to be needed to better define the cases in which the rule of mixtures can be accurately applied.

It has been shown that variations in the volume fraction of aligned phases can be produced by directional solidification of off-eutectic compositions, using high thermal gradients and relatively low growth rates [7-9]. In the work reported here, this technique was applied to the Al-Al₃Ni system in order to vary the volume fraction of Al₃Ni fibres and critically test the rule of mixtures, as applied to both the modulus and the ultimate tensile strength of the composite. In a parallel way, the effect of fibre morphology, produced at two different growth rates, on these properties was also evaluated.

2. Experimental procedure

An Al-30 wt % Ni master alloy was prepared from commercially pure aluminium (99.99%) and nickel (99.95%). Specific alloy compositions ranging

from 4.1 to 7.1 wt % nickel were prepared from the master alloy by melting under an argon atmosphere in an alumina crucible and then chillcasting in the form of 1.27 cm diameter rods. These rods were subsequently homogenized for 8 h at 823 K and swaged to final diameters of 0.317 and cm. The alloys were then inserted into graphite tubes 15 cm long with inner diameters just large enough to accommodate them. The chill-furnace used for growth at 1 cm h⁻¹ was previously described [9], and provided a gradient ranging from 473 to 553 K cm⁻¹. For a growth rate of 11 cm h⁻¹ and induction furnace and chill was used similar to that employed by Patarini *et al.* [10].

Reduced cross-sections were made on a lathe with dual-driven chucks to prevent twisting. Gauge sections, 1.25 cm in length and either 2.3 or 3.6 mm in diameter, were hand polished with $1 \,\mu$ m diamond paste prior to testing. A strain rate of 2% min⁻¹ was used, and the strain was measured with an extensometer.

Standard metallographic techniques were used to examine the composite microstructures. Etching was performed with dilute Keller's reagent and electropolishing with methanol-10% perchloric acid. Fibre volume fractions were measured by computerized image analysis using a Quantimet 720 and also by quantitative metallography as an additional check. Some specimens were deeply etched with a 3% hydrochloric acid solution to reveal the fibres. The chemical compositions were verified by wet chemical analysis.

3. Results and discussion

3.1 Fibre morphology

The Al₃Ni fibres of composites grown at 1 cm h^{-1} exhibited a blade-like morphology, which was the same over the range of 4.2 to 7.0 wt % Ni. The width of these fibres ranged from about 1.0 to 2.4 μ m. The transverse major axis of the fibres exhibited as many as three orientations within a single aluminium matrix grain (Fig. 1a). The longitudinal axis of most blade-like fibres showed only small $(<5^{\circ})$ angular deviations from the growth direction, with the exception of some fibres, juxtaposed to fibre-depleted regions, which deviated up to 30° from the growth direction (Fig. 1b). The longitudinal cross-sections of the ingots often displayed fibres with different etching characteristics, most probably because of different fibre-matrix orientation relationship. The blade-



Figure 1 Composite specimen grown at $1 \text{ cm} \text{h}^{-1}$, exhibiting blade-like fibre morphology. (a) Transverse section, \times 230; (b) Longitudinal section, \times 100.

like fibres, themselves, showed significant variations in cross-sectional dimensions along their length (Fig. 2). Wide fibres frequently branched into smaller fibres with transverse aspect ratios close to one (Fig. 2). Branching was usually confined to the plane containing the transverse major axis of the fibre.

Finely distributed aligned rod-like fibres were produced at a growth rate of 11 cm h⁻¹ (Fig. 3a). Fibre diameter varied from 0.4 to $0.8 \,\mu$ m. Fig. 3a showed some fibres with a transverse major axis preferentially oriented within the matrix grain.



Figure 2 (a) Al₃ Ni blade fibres extracted from specimen solidified at 1 cm h^{-1} and observed in transmitted light, $\times 150$, (b) scanning electron micrograph of a deeply etched longitudinal section of the same specimen, $\times 450$.



Figure 3 (a) Photomicrograph of a transverse section of a specimen grown at 11 cm h^{-1} , $\times 450$; (b) scanning electron micrograph of a deeply etched longitudinal section of the same specimen, $\times 1800$.

Extracted rod-fibres exhibited less branching than did blade-like fibres. A deeply etched specimen (Fig. 3b) shows axial alignment and more uniform fibre cross-sectional dimensions.

3.2. Fibre volume fraction

The volume fraction of Al_3Ni was calculated versus alloy composition using the lever rule and assuming a stoichiometric intermetallic of a 3.98 g cm⁻³ density [11] and no solubility of nickel in aluminium [12]. Comparison of calculated and experimentally measured volume fractions for composites with blade-like fibre-morphologies (Fig. 4) showed excellent agreement. Nickel solubility in the matrix is, therefore, quite low

Figure 4 Experimental and theoretical volume fraction $Al_3 Ni$ versus wt% nickel. Samples grown at 1 cm h^{-1} .



indeed and hence no appreciable solid solution or nickel precipitation hardening of the matrix would be expected.

3.3. Composite modulus

The load-elongation curves for composites with both fibre morphologies exhibited a typical transition, or knee, between complete elastic behaviour and the onset of yielding in the aluminium matrix (Fig. 5). The knee occurred between stress values of 34.5 and 73.8 MN m⁻² for both groups of composites. The modulus below the knee showed an increase with Al_3Ni volume fraction. Significant scatter of the data was attributed to seating of the extensometer upon loading and to inevitable inaccuracies in measuring the slope of a small line segment.

The application of the rule of mixtures to the elastic modulus yields:

$$E_{c} = E_{f}V_{f} + E_{m}(1 - V_{f})$$
(1)

where $E_{\rm e}$ is the elastic modulus of the composite, $E_{\rm f}$ that of Al₃Ni, $E_{\rm m}$ that of the matrix and $V_{\rm f}$ is the volume fraction of fibres. Above the knee of the load-elongation curve the matrix modulus can be replaced by the slope of the stress-strain curve



Figure 5 Typical load-elongation curve showing knee between completely elastic behaviour and elastic-plastic behaviour of $A1-A1_3$ Ni composites.

of pure aluminium at a specific composite strain ϵ_c :

$$E_{\mathbf{m}} = \left(\frac{\mathrm{d}\sigma_{\mathbf{A}\mathbf{I}}}{\mathrm{d}\epsilon_{\mathbf{A}\mathbf{I}}}\right)\epsilon_{\mathbf{c}} \,. \tag{2}$$

If the rate of strain hardening, $(d\sigma/d\epsilon)$, at ϵ_c is small compared to E_f the composite modulus can be given approximately by:

$$E_{\rm c} \approx E_{\rm f} V_{\rm f} \,.$$
 (3)



Figure 6 Tangent modulus measured just above the knee of the load-elongation curve for composites with rod-like fibres.

Experimentally measured values for the tangent modulus above the yield stress of the aluminium matrix for composites with rod-like fibres shows a linear increase with nickel content (Fig. 6). An extrapolation of the least squares fit of the data to to 25 at.% Ni (42 wt% Ni), where $V_f = 1$, yields a value of 146 GN m⁻² for the modulus of Al₃Ni according to Equation 3. This is in good agreement with the value of 131 to 152 GN m⁻² previously measured [2] in bend tests on extracted fibres. In composites with blade-like fibre morphologies a value of 131 GN m⁻² was obtained by extrapolation.

3.4. Composite tensile strength

The rule of mixtures when applied to tensile strength takes the form:

$$\sigma_{\mathbf{c}} = \sigma_{\mathbf{f}} V_{\mathbf{f}} + \sigma_{\mathbf{m}}^* (1 - V_{\mathbf{f}}) \tag{4}$$

where σ_c and σ_f are the tensile strengths of the composite and fibre, respectively, and σ_m^* is the

stress born by the matrix at the composite fracture strain. This rule predicts a linear strength dependence with volume fraction. A value of 2.9 GN m⁻² for the tensile strength of Al₃Ni fibres and 41.4 MN m⁻² for σ_m^* were previously established by Lemkey *et al.* [2] for the Al-Al₃Ni system and were used herein to establish a theoretical line for the rule of mixtures. This value of σ_m^* represents the strain hardened value of pure aluminium at a strain of about 2% and neglects dispersion hardening that may occur due to the presence of fine fibres.

Experimentally measured values of the strength of composites with both rod and blade fibre morphologies are compared with calculated values, using the rule of mixtures, in Fig. 7. Both morphologies show a linear increase in composite strength with fibre volume fraction. Strength for composites with blade fibres deviates significantly from the rule of mixtures, whereas that for composites with rod-like fibres shows a slight positive deviation from the rule, but nearly the same slope. Data for cellular composites [3] are also plotted in the same figure for comparison purposes.

If the rule of mixtures is fully applicable to composites with rod-like fibres (Fig. 7), an extrapolation of the composite tensile strength to $V_{\rm f} = 1$ would give the strength of Al₃Ni and an extrapolation to $V_{\rm f} = 0$ would give the stress in the matrix at the time of composite failure. Extrapolating the least-squares fit to the data for composites with rod-like fibres (Fig. 7) yields a value of 2.69 GN m⁻² for the tensile strength of Al_3 Ni fibres. This value agrees quite well with the 2.76 GNm⁻² value observed by Lemkey et al. [2] for the strongest fibres. Many of the tests which they conducted on extracted fibres showed a degradation in strength due to surface damage caused during extraction. The determination of fibre tensile strength from off-eutectic compositions circumverts this problem and provides a reasonably reliable value for composites with nearly uniform fibre diameters and whose matrices do not induce fibre failure.

The same extrapolation to pure aluminium $(V_{\rm f} =)$ yields for $\sigma_{\rm m}^*$ in Equation 4 a value of 86.3 MN m⁻². This value is about twice that previously assumed by Lemkey *et al.* [2]. A comparison between this value and the average value



Figure 7 Ultimate tensile strength versus wt % nickel or vol % Al_3 Ni. Rod fibre, blade fibre composites and cellular composites.

for the stress at which the aluminium matrix begins to yield (43.0 MN m^{-2}) suggests that the dispersion of rod-like fibres plays an important role in increasing both the yield point and subsequent strain hardening of the matrix, in agreement with similar observations made on eutectic composites [13].

The behaviour observed in Fig. 7 for composites with blade-like fibres does not obey the rule of mixtures. Although the variation in tensile strength with fibre volume fraction appears to be linear over the range studied, extrapolations to $V_{\rm f} = 0$ and $V_{\rm f} = 1$ predict an unreasonably high value for the matrix strength and far too low a value for the tensile strength of the fibres. This smaller slope could be attributed to different l/dratios^{*} as previously shown [14]. This geometric effect, however, does not change the intercept at $V_{\rm f} = 0$ and, therefore, cannot explain the experimental results for blade composites. Since at lower volume fractions the strength of blade composites approaches that of composites with rod-like fibres, it is quitepossible that blade composites begin to deviate from the rule of mixtures only above a volume percent fibres of about 5.

Over the range of compositions tested the composites with blade-like fibres show lower strengths and lower strains to failure than those with rod fibres. Composites with blades failed at strains of 1.0 to 1.6%, compared with 2.0 to 3.1% for those with rod fibres.

Smaller cross-sectional areas along the length of the blade fibres concentrate the stress, leading to fracture at lower composite strains. This is supported by the fracture behaviour of the composites with the two types of fibres. In composites with rod-like fibres the crack appears to have propagated along a plane of maximum shear, at about 45° to the tensile axis. In composites with blade-like fibres, on the other hand, the fracture surface is very rough and does not follow a plane of maximum shear stress. This latter behaviour could be attributed to the crack crossing regions where the blade-like fibres are discontinuous following the growth process or because of cracking during initial straining. Had the blade-like fibres been continuous and uniform in diameter along their length, their fracture behaviour would have been similar to that of rod-like fibres and composites containing them would probably obey the rule of mixtures.

4. Conclusions

(1) Off-eutectic compositions of $Al-Al_3Ni$ can be directionally solidified using high gradients and relatively low growth rates to produce variations in the volume fractions of aligned Al_3Ni fibres.

(2) The composite modulus at stresses above the yield strength of the aluminium matrix can be predicted by the rule of mixtures, $E_e = E_f V_f$, which assumes ideal plastic behaviour of the matrix. An extrapolation of these experimental data for composites with rod fibres gives a value of 146 GN m⁻² for the modulus of Al₃Ni.

(3) The composite tensile strength is strongly influenced by fibre morphology. Composites with uniform rod-like fibres grown at 11 cm h^{-1} behave according to the rule of mixtures. Composites with non-uniform blade fibres grown at 1 cm h^{-1} show lower strengths and do not behave according to the rule of mixtures.

(4) An extrapolation of data obtained from offeutectic compositions can be used to obtain the tensile strength of Al_3Ni fibres and the stress in the matrix at the time of composite failure. For composites with rod-like fibres these values are 2.69 GN m⁻² and 86.3 MN m⁻², respectively.

(5) Composites with blade-like fibres failed at lower strains than did those with more uniform rod-like fibres.

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