Investigation of the dynamic Young's modulus and vibration damping for cryomilled NiAI–AIN composites

A. WOLFENDEN, D. A. COAN

Mechanical Engineering Department, Texas A&M University, College Station, TX 77843, USA

M. G. HEBSUR NASA Lewis Research Center, Cleveland, OH 44135, USA E-mail: A1W3405@ACS.TAMU.edu

Measurements were made of the dynamic Young's modulus (stiffness) and damping for NiAI specimens containing varying amounts of aluminium nitride and prepared by cryomilling. Five specimens of NiAI–AIN were measured at room temperature, each specimen having a different percentage of AIN in the range 1%–30%. Further measurements were made on the 1% and 30% AIN specimens for the temperature range 22–495°C. At room temperature, the Young's modulus of the NiAI–AIN specimens decreased linearly with increasing percentage of AIN. The temperature dependence of Young's modulus for the 1% and 30% AIN specimens was found to be linear. These results were compared with those of typical nickel-based superalloys for a similar temperature range. The damping for all of the NiAI–AIN specimens was of the order of that for other intermetallic compounds ($10^{-3}-10^{-4}$). The study opened up the possibility of NiAI–AIN being a less-expensive and more easily produced alternative to nickel-based superalloys for high-temperature, high-stiffness applications. © *1998 Kluwer Academic Publishers*

1. Introduction

High-temperature, creep-resistant nickel aluminide (NiAl) composites have been produced by utilizing a reaction milling process called cryomilling. These composites have similar compressive properties to those of single-crystal nickel-based superalloys [1]. Research on new NiAl-AlN composites has suggested that these materials with high specific strength may lead to alternatives to expensive superalloys [2]. As an advanced ceramic, AlN is both interesting and attractive because of its good electric insulation and high thermal conductivity. The material has a relatively low density (3260 kg m^{-3}) and a Young's modulus (stiffness) of 35.7 GPa [3]. The present study focused on the measurement of dynamic Young's modulus and damping which are two of the most important properties required for design purposes. The measurement method used was the piezoelectric ultrasonic composite oscillator technique (PUCOT).

2. Materials preparation

High-intensity ball milling of NiAl powder in a liquid nitrogen-filled attritor chamber was performed at NASA Lewis Research Center to produce the NiAl– AlN composites. In this cryomilling process, the NiAl particles became fragmented and the near-surface regions became supersaturated with nitrogen. AlN precipitated within the nitrogen-rich layers during high-temperature powder consolidation operations such as hot isostatic pressing (HIPing) and extrusion [1, 2, 4]. The composites containing 1%, 5%, 20% and 30% AlN were consolidated by HIPing (see Fig. 1), while the NiAl-10%AlN composite was extruded. From these materials, specimens of dimensions 3 mm diameter \times 30 mm length were prepared for the PUCOT measurements. Fig. 1a and b show examples of the microstructure of the composites containing 1% and 30% AlN, respectively.

3. Density, modulus and damping measurements

The mass density (required for the determination of Young's modulus) of each specimen was measured by the Archimedes' technique. The dynamic Young's modulus and damping were measured using the PUCOT [5, 6]. This technique takes advantage of piezoelectric quartz crystals to excite longitudinal ultrasonic resonant stress waves in a specimen of appropriate resonant length. Two quartz crystals, one as a drive crystal and one as a gauge crystal, were joined end-to-end with cyanoacrylate glue and arranged in a holding jig (see Fig. 2). For the measurements at room temperature, the specimen was glued directly to the bottom of the gauge crystal; for the measurements





Figure 1 Microstructures of HIPed NiAl–AlN composites, containing (a) 1% AlN, and (b) 30% AlN.



Figure 2 Schematic drawing of the piezoelectric ultrasonic composite oscillator technique.

at elevated temperatures, the specimen was joined to the crystals via a spacer rod of fused quartz. The spacer rod was of appropriate resonant length for the test temperature. The joint between the spacer rod and the specimen was made with a ceramic glue. The reason for the spacer rod was to enable the drive and gauge crystals to be kept at room temperature while the specimen (and part of the spacer rod) were heated to the test temperature. A closed-loop oscillator drove the resonant system and maintained a constant gauge voltage and hence constant strain amplitude in the specimen. During a test, the values of the period of the resonant system, and of the drive and gauge voltages were recorded. These three values, along with the masses of the various components, and the length and density of the specimen, were used to determine the dynamic Young's modulus and the damping. Further details of the PUCOT are given elsewhere [5, 6].

4. Results and analysis

Table I contains the measured values of the mass density and Young's modulus for the specimens of different compositions at room temperature (21° C). Table II shows the values of Young's modulus for the 1% and 30% AlN specimens measured at elevated temperatures.

The density of the NiAl–AlN composites decreased linearly with increasing percentage of AlN and followed the rule of mixtures. This is shown in Figs 3 and 4. All of the specimens except the 10% AlN specimen were HIPed; the 10% AlN specimen was extruded. The difference in processing caused the value of the density for the 10% AlN specimen not to fit as well on the curve of density versus composition for all the specimens (Fig. 3). Hence, Fig. 4 contains only data for the HIPed specimens. It should be noted that the degree of fit in Fig. 4 ($R^2 = 0.9926$) is higher than that for Fig. 3 ($R^2 = 0.9462$).

TABLE I Measured values of mass density and Young's modulus for tests at room temperature

AlN (%)	Density (kg m ⁻³)	Young's modulus (GPa)
0 [3]	5871	234
1	5854	242
5	5769	278
10	5106	266
20	5267	182
30	5268	201
100 [6]	3260	36

TABLE II Measured values of Young's modulus as a function of temperature for the 1% and 30% AIN specimens

Temperature (°C)	Young's modulus (GPa)	
	1% AlN	30% AlN
21	242	201
100	237	
200	233	
350		180
361	225	190
425		181
450	218	
461		182
495	203	182



Figure 3 Density versus percentage of AlN for NiAl–AlN compounds: (\triangle) 1% AlN, (\blacksquare) 5% AlN, (\blacklozenge) 10% AlN, (\diamondsuit) 20% AlN, (\bigcirc) 30% AlN, (\blacklozenge) 100% AlN.



Figure 4 Density versus percentage of AlN for NiAl–AlN compounds, with the datum for the 10% AlN specimen removed, (\bigcirc) 0% AlN, (\blacktriangle) 1% AlN, (\blacksquare) 5% AlN, (\bigcirc) 20% AlN, (\triangle) 30% AlN, (\square) 100% AlN.

The dynamic Young's modulus was found to decrease with increasing percentage of AlN. This is demonstrated in Fig. 5 (all the data) and Fig. 6 (without the datum for the 10% AlN specimen). The degree of fit for the curve in Fig. 6 ($R^2 = 0.9269$) is somewhat higher than that for the curve in Fig. 5 ($R^2 = 0.9077$). The trend curve for Young's modulus follows the rule of mixtures.

The Young's modulus data for elevated temperatures are shown in Fig. 7. Young's modulus for the 1% and 30% AlN specimens decreased linearly with increasing temperature. For the 1% specimen, the value of R^2 is 0.8876 and the slope of the curve is -0.07 GPa K⁻¹. The corresponding values for the 30% specimen are $R^2 = 0.8061$ and slope = -0.04GPa K⁻¹. These trends follow those for most materials. The standard error of estimate for all the Young's modulus measurements was ± 4.13 GPa.

The Young's modulus data for both the 1% and 30% AlN specimens were compared with those for several nickel-based superalloys, as shown in Fig. 8. Six superalloys that had relatively high values of Young's modulus were chosen [7]. It is noted that the 1% AlN specimen has modulus values that are higher than all of those for the chosen superalloys at all temperatures investigated. The Young's modulus for the 1% specimen decreased at approximately the same rate as that



Figure 5 Dynamic Young's modulus versus percentage of AlN for NiAl–AlN compounds.



Figure 6 Dynamic Young's modulus versus percentage of AlN for NiAl–AlN compounds, with the datum for the 10% AlN specimen removed.



Figure 7 Dynamic Young's modulus versus temperature for the (\bullet) 1% and (\blacktriangle) 30% AlN specimens.

for all the superalloys. The 30% AlN specimen had values for Young's modulus that fell within a band defined by five of the six superalloys. The modulus for the 30% specimen also decreased at the same rate as that of the five superalloys. Both the 1% and 30% AlN specimens had values of modulus that were significantly greater than those of the nickel-based alloy MAR-M200 (DS). In summary, the mechanical property of Young's modulus for the 1% and 30% AlN specimens was seen to be comparable at elevated temperatures to that of several nickel-based superalloys.



Figure 8 Comparison of the temperature dependence of Young's modulus for the NiAl–AlN specimens with the dependence for six nickel-based alloys. (**●**) NiAl–AlN with 1% AlN; (**▲**) NiAl–AlN with 30% AlN; (**■**) nickel-based alloy 713C; (**♦**) nickel-based alloy 713LC; (**○**) nickel-based alloy IN-100; (**△**) nickel-based alloy IN-162; (**□**) nickel-based alloy MAR-M200; (**◊**) nickel-based alloy MAR-M200(DS). (**—**) linear (NiAl–AlN with 1% AlN); (---) linear (NiAl–AlN with 30% AlN); (---) linear (nickel-based alloy 713C); (**—**) linear (nickel-based alloy 713C); (**—**) linear (nickel-based alloy 713C); (**—**) linear (nickel-based alloy IN-162); (---) linear (nickel-based alloy IN-162); (---) linear (nickel-based alloy MAR-M200); (---) linear (nickel-based alloy MAR-M200) (DS)).

It is interesting to normalize the slopes of Young's modulus, E, versus temperature, T, by dividing the value of the slope by the value of Young's modulus at room temperature. The normalized slope is thus $(1/E_{\rm RT})(dE/dT)$. For the 1% and 30% AlN specimens the values of normalized slope were found to be -2.85×10^{-4} and $-2.18 \times 10^{-4} \text{ K}^{-1}$, respectively. From the literature, the corresponding values for many metals are in the range -4×10^{-4} to $-14 \times$ 10^{-4} K^{-1} [8], the values for oxides are in the range -1×10^{-4} to -2×10^{-4} K⁻¹ [9], and the values for NiAl and many nickel-based superalloys are near -6×10^{-4} K⁻¹ [10]. Thus, the normalized slopes for the 1% and 30% AIN specimens are near the high end of the range for ceramics and near the low end of the range for metals. This behaviour of the temperature dependence of the elastic modulus is a reflection of the mixed interatomic bonding in nickel aluminides, namely, partly covalent and partly metallic.

The mechanical damping of the NiAl–AlN composites was found to be of the order of 10^{-3} – 10^{-4} for room temperature and high temperatures. These relatively low values are due to the fact that NiAl is an intermetallic compound with a mixture of covalent and metallic bonding, making it behave somewhat like a ceramic with respect to damping.

5. Conclusion

The density of NiAl–AlN composites decreased from 5854 kg m⁻³ to 5268 kg m⁻³ as the percentage AlN increased from 1% to 30%, and followed the rule of mixtures. The dynamic Young's modulus measured at room temperature decreased from 242 GPa to 201 GPa for the same increase in percentage of AlN, and also followed the rule of mixtures. For the 1% and 30% NiAl specimens, Young's modulus decreased linearly from 242 GPa to 203 GPa and from 201 GPa to 182 GPa, respectively, as the temperature increased from 22 °C to 495 °C. The respective slopes dE/dT were -0.07 and -0.04 GPa K⁻¹. The mechanical damping was generally found to be of the order of that for other intermetallic compounds $(10^{-3}-10^{-4})$.

Young's modulus values for several nickel-based alloys were compared with those measured in this study. The present data demonstrated that the stiffness of the NiAl–AlN specimens was comparable to that of nickel-based superalloys even at elevated temperatures. It is possible that NiAl–AlN compounds may be utilized as a less-expensive alternative to nickel-based superalloys in high-temperature, highstiffness applications.

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