MICROHARDNESS AND ELASTIC MODULUS OF NANOCRYSTALLINE Al-Zr.*

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Abstract

An investigation of the mechanical properties of nanocrystalline Al-Zr alloy composites has been conducted via nanoindentation and Vickers microhardness experiments. The microhardness of the samples exhibits a four-fold increase over the concentration range of 0 - 30 wt.% Zr, from ~0.7 GPa to nearly 3 GPa. The aluminum grain size is found to be strongly correlated with the level of zirconium present in the samples, suggesting that the observed hardness increase can be attributed to the combined effects of alloying and grain size reduction. The elastic moduli of the nanocrystalline Al-Zr samples are determined to be similar to the modulus of coarse-grained aluminum and independent of zirconium content.

Introduction

There is a paucity of data in the literature about the mechanical properties of nanocrystalline materials produced by the inert-gas condensation (IGC) process, particularly for alloy and composite systems. This is largely due to limitations in sample size imposed by the IGC method, which typically yields only small quantities (tens of milligrams) of nanocrystalline powder that is consolidated into dime-sized disks. In order to elucidate the properties of gas-condensed nanocrystalline materials, therefore, indentation techniques and non-conventional testing methods must be employed. There is an additional motivation (besides sample size) for using indentation techniques to investigate the mechanical behavior of the nanocrystalline Al-Zr alloy composites in this study. Inherent variations in composition across the samples, which can be quantified using analytical electron microscopy techniques, can be exploited by indentation experiments that yield *local* mechanical properties data. In this paper, mechanical properties results as determined from indentation testing methods, in particular nanoindentation and Vickers microhardness experiments, are reported and discussed.

The purpose of this investigation was to determine the hardness and elastic modulus of the nanocrystalline Al-Zr samples in this study, and to consider the impact of microstructural variables, including chemical composition, grain size and porosity level, on these properties.

Experimental Details

The inert gas condensation (IGC) process [1,2] with electron beam heating [3], followed by uniaxial compression of the nanocrystalline powders at 1.4 GPa, was used to produce the samples in this study. The synthesis process is detailed elsewhere [4]. The compactions were done under high vacuum conditions at elevated temperatures of up to 100°C (0.4 T_m of aluminum).

The as-produced nanocrystalline samples underwent vibratory polishing to remove at least several microns of material, finishing with 0.05 μm alumina. Vickers microhardness data were obtained from a number of polished consolidated samples. A 100 g load was applied for 20 s for a total of 10-20 measurements per sample. Nanoindentation experiments were performed on several nanocrystalline samples and a coarse-grained aluminum standard using a load- and depth-sensing indentation tester [5]. A triangular pyramidal-shaped diamond indenter was driven into the samples at a rate of 3 nm/sec to maximum loads ranging from 1/6 g to 2 g. Approximately thirty load versus displacement curves were obtained for each sample from load relaxation, creep and indentation rate change experiments. From these data hardnesses and elastic moduli were calculated as described elsewhere [6].

Energy dispersive spectroscopy (EDS) data were then obtained during scanning electron microscope (SEM) examinations from small regions containing individual indents. The EDS data were collected at 10 kV and a 40° take-off angle. Analysis was done with standards using the ZAF technique [7] and a commercial program [8]. Density data were obtained using a technique based on Archimedes' principle as described in [9]. Two or more measurements were made for the mass of the sample and standard in air and ethyl phalate and the resulting density values were averaged. An aluminum standard was used to obtain values for the density of each sample in absolute terms (g/cm^3), and calculations based on the zirconium and oxygen concentrations in each sample were used to convert these numbers to more meaningful relative values (% of theoretical), as described previously [10].

From several samples 3 mm disks were cut, mechanically thinned and then electropolished [11] for transmission electron microscope examinations.

Results and Discussion

As described previously [10], the microstructures of the samples consist of nanocrystalline aluminum, the majority phase, and nanometer-sized particles of other phases, including the

cubic Al_3Zr structure. Recent experiments [12] suggest that some portion of the zirconium in the as-produced samples is also in solid solution, probably at the grain boundaries, given the extremely low lattice solubility of zirconium in aluminum [13] and the large volume fraction of grain boundaries in these materials.

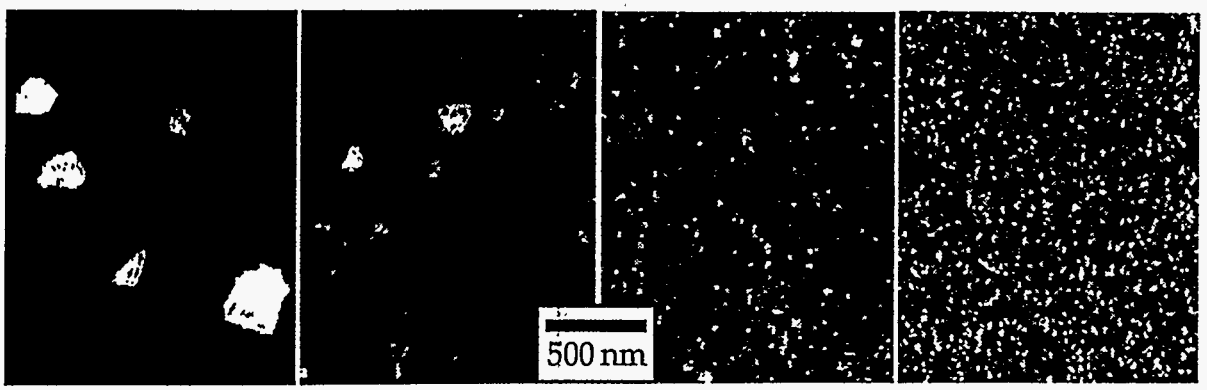

Figure 1. Dark field micrographs of aluminum grains in several samples consolidated at 100°C which show regions containing (a) no zirconium; (b) 0.4 wt.% Zr; (c) 2.0 wt.% Zr; and (d) 11.0 wt.% Zr.

Transmission electron microscopy studies indicate that the aluminum grains are stabilized at smaller grain sizes with increasing amounts of zirconium. This is evidenced in Figure 1, in which a series of dark field micrographs of aluminum grains from several as-produced samples, all consolidated at 100°C (0.4 T_m of aluminum) are shown. Because the compaction temperature is a significant fraction of the melting temperature of aluminum, it is believed that significant grain growth (up to several hundred nanometers) occurs during the consolidation process in samples containing < ~2 wt.% zirconium.

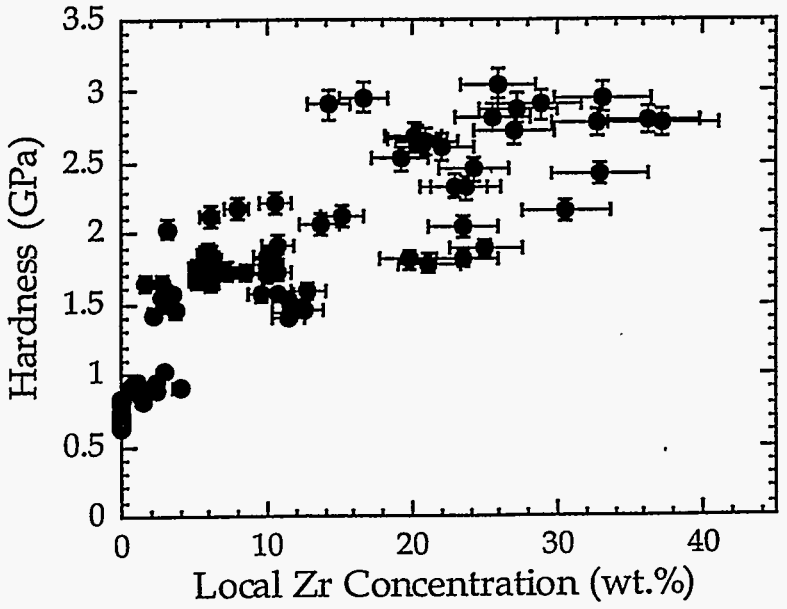

Figure 2. Microhardness as a function of local zirconium concentration, where each data point represents a single indentation. Data from several samples are shown.

The Vickers microhardness of the nanocrystalline Al-Zr samples is found to increase with zirconium content as shown in Figure 2. Each data point in the plot indicates the hardness and zirconium concentration of a single indentation, and data from a number of samples are shown. The hardness values range from ~0.7 GPa in regions of samples containing no zirconium, to nearly 3 GPa in regions of ~30 wt.% Zr. The correlation between zirconium

concentration and grain size established above suggests that the increased microhardness observed in the nanocrystalline Al-Zr samples is due to the combined effects of alloying and grain size reduction. Microhardness is found to be essentially independent of porosity level (the complement of which is the percent of theoretical density) when the average zirconium levels of the samples are considered, as indicated in Figure 3. In a previous study of the microhardness of nanocrystalline Al-Zr [10], zirconium concentration was found to be less important in determining the microhardness than the grain size and porosity level; however, in that study a smaller range of zirconium concentrations was considered (up to ~10 wt.% Zr) and the samples were not all consolidated at 100°C.

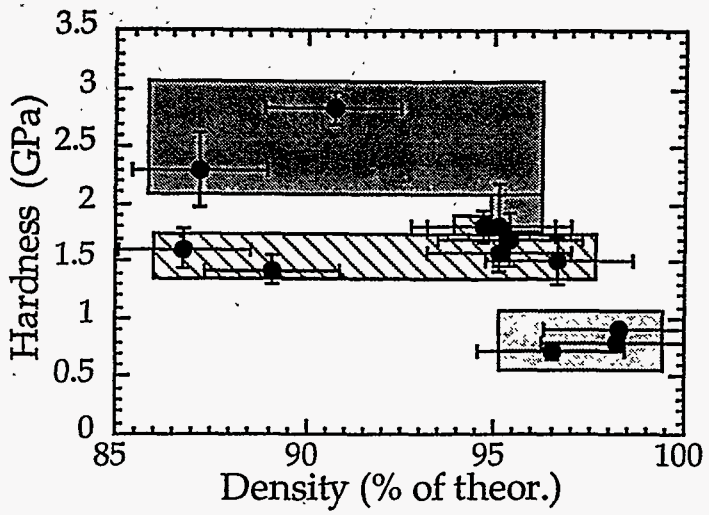

Figure 3. Average hardness as a function of density for a number of samples. The shaded regions indicate the average zirconium concentrations of the samples: ▨, < 2 wt.% Zr; ▧, 2 wt.% < Zr < 10 wt.%; ▩, 10 wt.% < Zr < 25 wt.% Zr.

Average values of the hardness and elastic modulus determined from nanoindentation are given in Table I for four nanocrystalline samples A-D, and coarse-grained aluminum sheet. Vickers microhardness data are shown as well for comparison. The hardness data, shown in the second and third columns, indicate that there is good agreement between the results of the nanoindentation and Vickers microhardness experiments. (The difference between the Vickers and nanoindentation hardness values in the case of the large-grained aluminum sample can be attributed to an indentation size effect.) In the former case, the indentation areas used in the hardness calculations were derived from load versus displacement data, while in the latter case the areas were determined from direct optical measurements of the indents. The modulus results given in the fourth column demonstrate that within the standard deviations of the measurements the elastic moduli of the nanocrystalline samples are similar to the modulus of coarse-grained aluminum. It should also be noted that the modulus of the coarse-grained aluminum sample as determined by nanoindentation, 68 ± 7 GPa, is consistent with reference values for aluminum (69 GPa) [14]. The modulus results presented here are consistent with a number of recent studies that show similar modulus values for nanocrystalline metals and their coarse-grained counterparts [15-17].

Although there have been a number of earlier reports of reduced moduli in nanocrystalline materials produced by the inert gas condensation process [18,19], it appears that the reported reductions can be attributed to either relatively high porosity levels or inadequate testing procedures. The porosity levels of all of the samples in this nanoindentation study were < 5%, with the exception of sample D (~13%). The presence of zirconium in the samples (the average zirconium concentrations of the four samples ranged from < 1 to 8.9 wt.% Zr) might be expected to result in slightly elevated moduli compared to that of pure aluminum; however, no compositional trends are observed in the data, in contrast to the hardness results discussed above. This is illustrated in Figure 4, where the elastic modulus

is plotted as a function of local zirconium concentration. Each data point corresponds to a single indentation, and data from the four nanocrystalline samples are plotted.

Table I. Nanoindentation Elastic Modulus (E) and Hardness (Hn) Values.

Sample	Hv (GPa)	Hn(0.9272)* (GPa)	E (GPa)
A	0.90 ± 0.06	0.86 ± 0.28	66 ± 9
B	1.53 ± 0.21	1.53 ± 0.34	71 ± 7
C	1.57 ± 0.13	1.59 ± 0.40	69 ± 3
D	1.69 ± 0.19	1.75 ± 0.53	71 ± 3
c-g Al	0.29 ± 0.01	0.43 ± 0.05	68 ± 7

Errors given represent standard deviations.
* In Vickers hardness tests the contact area of the indent is measured; in nanoindentation the projected contact area is determined. The geometrical factor 0.9272 brings the hardness values into coincidence.

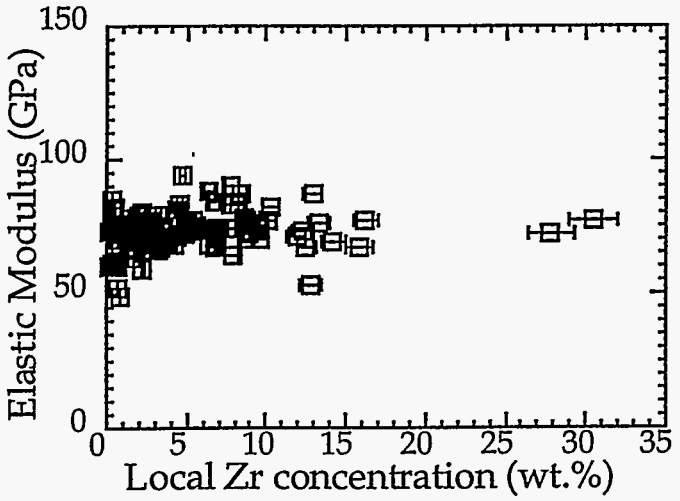

Figure 4. Elastic modulus as a function of local Zr content. Each data point represents a single indentation and data from nanocrystalline samples A-D are shown.

Conclusions

In this investigation of nanocrystalline Al-Zr alloys produced by the inert gas condensation process, hardnesses and elastic moduli have been determined by nanoindentation and Vickers microhardness experiments. The impact of microstructural variables, including chemical composition, grain size and porosity level, on these properties has been considered. The microhardness of the samples is found to increase fourfold over the concentration range of 0 - 30 wt.% Zr, from ~0.7 GPa to nearly 3 GPa. The aluminum grain size is found to be strongly correlated with the level of zirconium present in the samples, suggesting that the hardness increase observed may be attributed to the combined effects of alloying and grain size reduction. The elastic moduli of the nanocrystalline Al-Zr samples are determined to be similar to that of coarse-grained aluminum, and appear to be independent of local composition.

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