Production of Nanograined Intermetallics using High-pressure Torsion

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Formation of intermetallics is generally feasible at high temperatures when the lattice diffusion is fast enough to form the ordered phases. This study shows that nanograined intermetallics are formed at a low temperature as 573 K in Al- 25 mol% Ni, Al- 50 mol.% Ni and Al- 50 mol% Ti powder mixtures through powder consolidation using high-pressure torsion (HPT). For the three compositions, the hardness gradually increases with straining but saturates to the levels as high as 550-920 Hv. In addition to the high hardness, the TiAl material exhibits high yield strength as ~3 GPa with good ductility as ~23%, when they are examined by micropillar compression tests. X-ray diffraction analysis and high-resolution transmission electron microscopy reveal that the significant increase in hardness and strength is due to the formation of nanograined intermetallics such as Al₃Ni, Al₃Ni₂, TiAl₃, TiAl₂ and TiAl with average grain sizes of 20-40 nm.

Keywords: severe plastic deformation (SPD), ordering, phase transformation, micropillar, AlNi, TiAl, aluminide

1. Introduction

High-pressure torsion (HPT) was first introduced by Bridgman in 1935 to investigate the mechanical behavior and phase transformations in materials under high pressure and concurrent torsional straining¹. The principle of the HPT processing is that a sample, in the form of disc or ring, is placed between two anvils which are rotated with respect to each other under application of compressive pressure, P , to create torsional strain, γ , in the sample².

$$\gamma = \frac{2\pi r N}{t} \quad (1)$$

where r is the distance from the center of disc (or ring), N is the number of turns and t is the thickness of disc (or ring). In 1991, Valiev et al. reported the significance of grain refinement to the submicrometer and nanometer levels by HPT³. Over the last two decades, considerable interest has developed in processing materials through the application of HPT as a severe plastic deformation method not only for grain refinement⁴⁻⁶, but also for several other applications such as attainment of ultrahigh strength and high ductility⁷⁻⁹, attainment of high strength and high electrical conductivity¹⁰, achievement of high strength and high biocompatibility¹¹⁻¹³, improvement of Tribocorrosion resistance¹⁴, improvement of wear resistance¹⁵, improvement of hydrogen storage capability¹⁶⁻¹⁸, achievement of photoluminescence effect¹⁹, controlling the allotropic phase transformations¹⁹⁻²², consolidation of machining chips^{23,24}, consolidation of metallic powders²⁵⁻²⁷, production

of supersaturated alloys^{28,29}, and improvement of several other multifunctionalities³⁰⁻³². Although most of these works are focused on HPT processing using bulk samples, consolidation of powders using HPT has recently received much attention²³⁻³⁰.

The HPT method was recently applied for production of nanostructured intermetallics with ultrahigh strength and high ductility from their elemental constituents^{33,34}. The method was applied to powder mixtures of the Al-Ni and Al-Ti systems and it was found that in addition to powders consolidation and grain refinement, nanograined intermetallics were formed. This paper reports summary from the earlier study with additional results on the production of the nanograined AlNi and TiAl intermetallics^{33,34} and an extended application of the principle to an Al₃Ni production from the Al and Ni elemental powders.

2. Experimental Procedures

Pure Al (99.99%), Ni (99.99%) and Ti (99.9%) were received in the form of micropowders with particle sizes less than 75 μm , 50 μm and 150 μm , respectively. Powder mixtures of Al- 25% Ni, Al- 50% Ni and Al- 50% Ti were prepared by mechanical agitation (all compositions are in mol%). HPT was conducted at 573 K to consolidate the powder mixtures to discs with 10 mm diameter and 0.8 mm thickness under a pressure of $P = 6$ GPa. Shear strain was introduced through rotations for either $N = 3, 10, 25, 50$ or 120 turns with a rotation speed of 1 rpm.

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The HPT-processed discs were first polished to a mirror-like surface and Vickers microhardness was measured with an applied load of 200 g for 15 s along the radii at 8 different radial directions. Second, X-ray diffraction (XRD) analysis was performed using the Cu K α radiation in a scanning step of 0.01° and a scanning speed of 0.5 °/min. Third, for transmission electron microscopy (TEM), discs with 3 mm in diameter were cut from the HPT-processed discs at 3.5 mm away from the center. The 3 mm discs were ground to a thickness of 0.15 mm and further thinned for TEM with a solution of 10% H₂SO₄, 10% HNO₃ and 80% CH₃OH at 263 K under an applied voltage of 18 V for the Al-Ni samples and using a solution of 5% HClO₄, 25% C₃H₇(CH₂)₂CH₂OH and 70% CH₃OH at 263 K under an applied voltage of 15 V for the Al-Ti samples. TEM was performed under a voltage of either 200 kV or 300 kV for microstructural observation and for recording selected-area electron diffraction (SAED) patterns. Fourth, the mirror-like surface of the HPT-processed samples was examined using scanning electron microscopy (SEM) under an applied voltage of 15 kV to analyze the formation of micropores during HPT. Fifth, square-shaped micropillars with a side length of ~4 μ m and a height of ~12 μ m were prepared from the discs at ~4 mm away from the center using focused ion beam (FIB) technique so that the side surfaces of the pillars become perpendicular to the disc surface³⁴. Compression test was conducted on the micropillars using a microhardness testing machine equipped with a flat diamond tip with a diameter of 20 μ m at a nominal stress rate of 10 MPa/s, which corresponds to an initial strain rate of 10⁻⁴ s⁻¹. Sixth, the micropillars were observed by SEM under an applied voltage of 15 kV.

3. Results and Discussion

Figure 1 shows the hardness variation with the distance from the disc center after processing by HPT for various turns on the (a) Al- 25% Ni, (b) Al- 50% Ni and (c) Al-50% Ti samples. The microhardness increases with an increasing number of turns and an increasing distance from the disc center. The difference in the hardness behavior arises because the magnitude of strain created through HPT increases with increasing the turn and the distance from the disc center as given by Equation 1. The saturation of the hardness level is attained in the disc samples after 50 turns in the Al- 25% Ni and after 10 turns in the Al- 50% Ni and Al- 50% Ti, indicating that increasing the atomic fraction of the second element can accelerate the hardening during HPT. The hardness levels at the saturation, 550-920 Hv, exceeds those of most HPT-processed metals and alloys reported thus far²⁻⁷.

XRD profiles are shown in Figure 2 for the powder mixtures of (a) Al- 25% Al, (b) Al- 50% Ni and (c) Al-50% Ti and for the corresponding compositions of discs consolidated by HPT for various turns. A close examination of Figure 2 indicates the four important points. First, the Al₃Ni intermetallic is formed in the Al-25% Ni after processing the powders by HPT, but a certain fraction of Al₃Ni₂ intermetallic are detected at large strains. The formation of intermetallics are controlled by atomic

diffusion and it is well documented that the diffusivity can strongly be enhanced during severe plastic deformation because of the presence of large fractions of high-angle grain boundaries^{35,36} as well as because of supersaturation of vacancies³⁷. Second, the Al₃Ni intermetallics are formed at an early stage of straining in the Al- 50% Ni sample, but they transform to Al₃Ni₂ intermetallic at large strains. Since the diffusion of Ni in Al is faster than the diffusion of Al in Ni³⁸, the Ni atoms diffuse to the Al matrix and form the Al-rich Al₃Ni intermetallics which then transforms to the Al₃Ni₂ intermetallics with further diffusion of Ni atoms. Third, large fractions of TiAl₃, TiAl₂ and TiAl intermetallics are formed in the Al- 50% Ti and their fraction increases with increasing strain. The TiAl₃ cannot be detected at the steady state because the diffusivity of Ti in TiAl₃ is rather fast³⁸ and it transforms to the TiAl₂ and TiAl intermetallics. Fourth, the

Figure 1. Vickers microhardness plotted against distance from disc center for (a) Al- 25% Ni, (b) Al- 50% Ni and (c) Al- 50% Ti samples processed by HPT for various numbers of turns.

full width at half maximum in the XRD patterns increases significantly with torsional straining using HPT, indicating the occurrence of lattice strains, dislocations generation and grain fragmentation during the HPT processing.

TEM micrographs including SAED patterns are shown in Figure 3 for the Al- 25% Ni samples after HPT, where

Figure 2. XRD profiles for (a) Al- 25% Ni, (b) Al- 50% Ni and (c) Al- 50% Ti samples processed by HPT for various numbers of turns.

the bright-field images are on the left, the SAED patterns are in the inset at the center and the dark-field images taken with the diffracted beams indicated by the arrows in the SAED patterns are on the right. The micrographs and the corresponding SAED patterns were taken from four samples subjected to different numbers of turns: (a) $N = 3$, (b) $N = 10$, (c) $N = 25$ and (d) $N = 50$.

Observation shows that the microstructure corresponding to Figure 3a consists of large grains with an average grain size of ~ 2500 nm. Although grains containing high dislocation density are locally visible within the microstructures as marked **A**, few dislocations are visible within most of the grains with the grain boundaries well-defined in (a). It is noted that these microstructural features are typical of microstructures after processing by HPT at high homologous temperatures^{4,10}. With increasing the shear stain, the grains are refined to the submicrometer level as in Figure 3b and many Al_3Ni nanograins as marked **B** are visible within the microstructures. In Figure 3c the grain size is reduced to the

Figure 3. TEM bright-field images (left), SAED patterns (center) and dark-field images taken with diffracted beams indicated by arrows in SAED patterns (right) for Al- 25% Ni samples processed for (a) 3, (b) 10, (c) 25 and (d) 50 turns.

Figure 5. TEM micrographs of Al- 50% Ti sample after HPT processing for 50 turns. (a) bright-field image and corresponding SAED pattern, (b) dark-field image taken with diffracted beams indicated by arrow in SAED patterns, (c) high resolution image and corresponding FFT analysis (d) reconstructed lattice image of square region in (c) after inverse FFT analysis, showing presence of $\{111\}\langle 110 \rangle$ edge dislocation.

compression test was used in this study because of several reasons. First, despite many papers regarding the tensile properties of HPT-processed materials^{2,7,9,12,16,23,26,27}, there are limited reports regarding their compression properties³⁴. Second, since some fractions of micropores are formed during consolidation by HPT^{33,34}, the micropillar was used to minimize the effect of micropores on the mechanical properties. Third, the micropillars are appropriate to investigate the deformation mechanism⁴³. Figure 6 shows representative stress-strain curves of the samples. The sample after HPT processing but before annealing, which consists of certain fractions of Ti, TiAl_2 and TiAl , exhibits a high yield strength as ~ 1.7 GPa but a limited plasticity as 2%. The sample after HPT processing and annealing, which is composed only of TiAl , exhibits a plasticity as high as $\sim 23\%$. The yield strength is also significantly enhanced to the level as high as ~ 3 GPa and this strength is 4-10 times higher than those for micropillars of TiAl single crystals⁴³. It should be noted that the sample after annealing had a bimodal microstructure composed of nanograins and submicrometer grains with an average grain size of ~ 100 nm with a high density of nanotwins with an average twin width of ~ 9 nm.

Figure 7 shows the TiAl micropillars (a) before compression test and (b) after compression test. It is evident that the pillar is deformed plastically with an estimated uniform plasticity of $\sim 19\%$ which is well consistent with the compression test. The high uniform plasticity may be attributed to the two microstructural features: one for bimodal microstructure formation composed of nanograins and submicrometer grains and the other for nanotwins formation as discussed earlier³⁴.

Figure 6. Nominal stress versus nominal strain curves for Al- 50% Ti samples after HPT processing for 50 turns and after HPT processing and annealing at 873 K for 1 hour. Micropillar compression tests were carried out at nominal stress rate of 10 MPa/s, corresponding to initial strain rate of 10^{-4} s^{-1} .

Figure 7. Appearance of pillars for Al- 50% Ti sample processed by HPT for 50 turns. (a) before compression and (b) after compression observed by SEM.

4. Conclusions

- Micropowder mixtures of Al- 25% Ni, Al- 50% Ni and Al- 50% Ti were consolidated by HPT at a temperature of 573 K;
- Large fractions of intermetallics such as Al_3Ni , Al_3Ni_2 , TiAl_3 , TiAl_2 and TiAl were formed because of enhanced diffusivity;
- Along with the formation of intermetallics, the grain size was reduced to 20-40 nm and the hardness was increased to 550-920 Hv;
- The compression strength was ~ 1.7 GPa with $\sim 2\%$ ductility in the Al- 50% Ti samples processed by HPT, but increased to ~ 3 GPa with the ductility as high as $\sim 23\%$ after subsequent annealing.

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