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 Al\textsubscript{2}Ni, Al\textsubscript{3}Ni\textsubscript{2}, TiAl\textsubscript{13}, TiAl\textsubscript{2} 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 straining\textsuperscript{1}. 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, \( \gamma \), in the sample:\textsuperscript{2}

\[ \gamma = \frac{2\pi r N}{t} \]  \hspace{1cm} (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 HPT\textsuperscript{3}. 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 refinement\textsuperscript{4,5}, but also for several other applications such as attainment of ultrahigh strength and high ductility\textsuperscript{6,7}, attainment of high strength and high electrical conductivity\textsuperscript{8}, achievement of high strength and high biocompatibility\textsuperscript{9,10}, improvement of Tribocorrosion resistance\textsuperscript{11}, improvement of wear resistance\textsuperscript{12}, improvement of hydrogen storage capability\textsuperscript{13,14}, achievement of photoluminescence effect\textsuperscript{15}, controlling the allotropic phase transformations\textsuperscript{16}, consolidation of machining chips\textsuperscript{17,18}, consolidation of metallic powders\textsuperscript{19,20}, production of supersaturated alloys\textsuperscript{21,22}, and improvement of several other multifunctionalities\textsuperscript{23,24}. Although most of these works are focused on HPT processing using bulk samples, consolidation of powders using HPT has recently received much attention\textsuperscript{25,26}.

The HPT method was recently applied for production of nanostructured intermetallics with ultrahigh strength and high ductility from their elemental constituents\textsuperscript{27,28}. 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\textsuperscript{29,30} and an extended application of the principle to Al-Ni 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 \( \mu \)m, 50 \( \mu \)m and 150 \( \mu \)m, 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 Kα 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% C₃H₅(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 µm and a height of ~12 µm 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 surface. Compression test was conducted on the micropillars using a microhardness testing machine equipped with a flat diamond tip with a diameter of 20 µm 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 Al₅Ni intermetallic is formed in the Al-25% Ni after processing the powders by HPT, but a certain fraction of Al₅Ni₁ 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 boundaries³⁵,³⁶ as well as because of supersaturation of vacancies³⁷. Second, the Al₅Ni intermetallics are formed at an early stage of straining in the Al-50% Ni sample, but they transform to Al₅Ni₁ 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 Al₅Ni intermetallics which then transforms to the Al₅Ni₁ intermetallics with further diffusion of Ni atoms. Third, large fractions of TiAl₅, TiAl₁ and TiAl intermetallics are formed in the Al-50% Ti and their fraction increases with increasing strain. The TiAl₁ cannot be detected at the steady state because the diffusivity of Ti in TiAl₁, is rather fast³⁸ and it transforms to the TiAl₁ 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 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. With increasing the shear strain, the grains are refined to the submicrometer level as in Figure 3b and many Al$_3$Ni nanograins as marked B are visible within the microstructures. In Figure 3c the grain size is reduced to the

![Figure 2](image)

**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.

![Figure 3](image)

**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.
nanometer level and the ill-defined grain boundaries increase the misorientation angles because the SAED analysis now exhibits a ring pattern. In Figure 3d, the grain size reaches ~40 nm and the grain boundaries appear to be better defined. The ring pattern from the SAED analysis indicates that the nanogranites are separated by high angles of misorientations at the steady state.

The grain size of ~40 nm is much smaller than those of the HPT-processed pure metals\(^4,10,16,26\) and many alloys\(^3,5,6,9\), but well comparable with those of HPT-processed intermetallics\(^39,40\), ceramics\(^21\), lattice softened alloys\(^8\) and semi-metals such as Si\(^19\). The formation of nanogranites can be attributed to two main reasons: first, the presence of a second phase blocks the dislocations motion and grain boundaries movement, and second, the in-situ formed intermetallics have strong covalent bonding. For the latter, it was reported that the grain size in materials with covalent bonding is significantly reduced to the nanometer level by HPT\(^9\). The application of HPT to intermetallics as well as other materials with covalent bonding results in formation of a heterogeneous microstructure composed of nanogranites and submicrometer grains\(^39,40\), whereas the grain size distribution is reasonably uniform after in situ production with HPT\(^33\). This is an important advantage of in-situ production of nanograined intermetallics by HPT.

High-resolution TEM images and corresponding diffractograms obtained by fast Fourier transform (FFT) analyses are shown in Figure 4 for the Al- 50% Ni sample processed by HPT for 50 turns. The FFT analyses show that (a), (b) and (c) correspond to Ni, Al\(_3\)Ni and Al\(_3\)Ni\(_2\), respectively. This characterization is well consistent with the XRD analyses. The average grain size for this sample is ~30 nm which is slightly smaller than the steady-state grain size of the HPT-processed Al- 25% Ni sample. It should be noted that this sample after annealing at 673 K transform to ~100% AlNi intermetallic with an average grain size of ~50 nm\(^33\).

Microstructures are shown in Figure 5 for the Al- 50% Ti sample processed for 50 turns, where (a) is a TEM bright-field image including the diffracted beams indicated by the arrow in the SAED pattern, (c) is a high resolution image and the corresponding diffractogram, and (d) is a reconstructed lattice images of the square region in (c) obtained by inverse FFT, which corresponds to either Al or TiAl. Note that the micrographs were taken on the sample at the steady state where the hardness remains unchanged with straining. The TEM characterization indicates several important points.

First, the bright- and dark-field images show that the nanogranites form after HPT processing with an average grain size of ~20 nm. It should be noted that this sample transforms to ~100% TiAl after annealing at 873 K with an average grain size of ~100 nm with a large fraction of nanotwins\(^34\). Second, the SAED pattern exhibits a complete form of rings, indicating that the microstructure consists of very small grains having high angles of misorientations. Third, the high-resolution image also shows the formation of nanogranites. Fourth, examination of the lattice image clearly shows that there is at least one edge dislocation in the interior of the grain. Considering the grain size of ~20 nm, an estimation of the minimum dislocation density results in \(3.2 \times 10^{15} \text{ m}^{-2}\), provided that at least a single dislocation exists in each nanograin. It turns out that such a high dislocation density within the nanogranites, which is consistent with the peak broadening in the XRD patterns, is comparable to that in HPT-processed pure metals\(^42\), alloys\(^6\) and ceramics\(^35\).

Two samples after processing by HPT and after the HPT processing with subsequent annealing were subjected to micropillar compression testing at room temperature with a pillar size of ~4 x 4 x 12 \(\mu\text{m}^3\). The micropillar
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$_3$Ni, Al$_3$Ni$_2$, 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 ~2% ductility in the Al-50% Ti samples processed by HPT, but increased to ~3 GPa with the ductility as high as ~23% after subsequent annealing.

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