Optimization of high pressure torsion processing for fabrication of the Al-Nb hybrid system

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Abstract. A hybrid composite material fabricated from the Al-Nb system using high-pressure torsion (HPT) up to 30 turns has been studied in the present work. To fabricate the composite, deformation of a three-layer Al-Nb-Al package was carried out at room temperature on Bridgman anvils with grooves under a pressure of 5 GPa at N=10, 25 and 30 revolutions, at a strain rate of ω=1 and 2 rpm. Initial disc diameters from pure metals and HPT conditions were experimentally optimized to obtain monolithic and defect-free composite samples. The most intensive fragmentation and stirring of niobium in the aluminium matrix was observed if diameter of aluminium discs was 10 mm and deformation conditions N=25 and 30 revolutions and ω=2 rpm were applied. Three microstructural zones were observed after HPT under optimal conditions: the central zone with wide curved layers of niobium in aluminium, the mid-radius zone with finely dispersed layered structure, and periphery with a uniform distribution of niobium in the aluminium matrix. It was shown that HPT led to occurrence of strain-induced ageing resulting in formation of the intermetallic Al3Nb phase. The microhardness measured along the diameter of the obtained composite materials changed nonmonotonically depending on the produced structure (microstructural zone).

1. Introduction

To create aluminum-matrix composite, from standpoint of approximation of the interaction system of the reinforcing phase and the Al matrix to a thermodynamically stable one, various methods based on the in-situ principle are actively used [1-3]. In this case, hardening intermetallic phases playing the role of reinforcements are formed in the Al matrix due to their nucleation and growth. One of the methods is severe plastic deformation by high-pressure torsion (HPT) [4]. Different layered metal-matrix composites have been recently fabricated via HPT of a stacking of discs of two dissimilar metals such as Al-Mg [5], Al-Cu [6], Ag-Cu [7]. In this case, hybrid materials having a unique multilevel structure resulted from the occurrence of diffusion processes are formed. An aluminum-matrix composite based on the Al-Nb system is of great interest to create the hybrid composite material, because the melting point of niobium is 2.5 times higher than that of pure Al and the solid state solubility of Nb in Al is near zero. This will expectedly lead to the greatest contribution of the hardening intermetallic phase(s) synthesized during HPT to the strength and other mechanical properties.

The aim of the present work was to access the feasibility of fabrication in-situ of the hybrid composite materials based on the Al-Nb system via HPT.
2. Materials and methods
The initial materials were Al (with purity 99.5%) and Nb (with purity 99%). The diameters of the Al discs were 6, 7, 8, 10, and 12 mm, and the Nb discs were 12 mm in diameter. The thickness of the Al discs was in the range of 0.3-1.2 mm and the Nb discs were 0.5 mm in thickness. The aluminium and niobium discs were laid in the three-layer Al-Nb-Al package.

HPT of the Al-Nb-Al package was carried out at the room temperature (RT) on Bridgman anvils under a pressure of 5 GPa at N=10, 25, and 30 revolutions, at a rotation speed of ω=1 and 2 rpm. The final size of the aluminum-matrix composite samples obtained via HPT was Ø12 mm × 0.6 mm.

The microstructure examination and microhardness measurements were carried out from the cross sections of the composite samples. Microstructural observations were conducted using a Tescan Mira 3LMH scanning electron microscope in the backscattering electron (BSE) mode. Transmission electron microscopic (TEM) studies were performed on a JEM 2100 Plus microscope at 200 kV accelerating voltage. The selected area diffraction pattern was obtained from a 2 μm² area. The X-ray diffraction analysis was performed for the cross section of the disc using a high-resolution Rigaku Ultima IV X-Ray diffraction system and Cu-Kα radiation. Microhardness was measured by the Vickers method on an AFFRI DM8A microhardness tester with 10 g indentation load.

3. Results and discussion
As has been shown elsewhere [6], the formation of monolithic aluminum-matrix composite of three-layer Al-Cu-Al packets by HPT took place at an optimal pressure of about 5 GPa, ω=1 rpm and N=10. In this case, the diameter of the Al and Cu discs was 12 mm, the initial thickness was about 0.5 mm. However, the use of the same diameters of the Al and Nb discs and the same HPT conditions, intensive stirring of the layers did not occur (figure 1). The sample was retained layered and only some fragmentation of layers was detected at periphery of the sample (figure 1). Therefore, the varied initial diameters of Al discs (6, 7, 8, and 10 mm) were used for HPT of the Al-Nb-Al packages. In these experiments, the geometry of the Nb discs remained unchangeable. SEM observations of the composite samples produced by HPT (N=10, ω=1 rpm) showed that the most intensive fragmentation and stirring of niobium in the aluminum matrix were achieved with an Al disc diameter of 10 mm (figure 1). So, the next variations of HPT conditions were fulfilled using the Al discs of 10 mm in diameter.

Increased deformation rate due to an increase in the number of sliding systems involved in deformation [8], led to a more significant fragmentation and a better niobium distribution within the sample. The Al-Nb-Al samples were deformed to 25 and 30 revolutions for further microstructure refinement and mixing of the Al and Nb layers. In doing so, monolithic deformed samples free of any visible cracks and chips were obtained. In the samples deformed to 25 and 30 revolutions the intensive fragmentation and mixing of the Al and Nb layers was attained practically in the entire volume of the deformed samples (figure 1) that is consistent with ref. [9].

The HPT of the Al-Nb-Al samples at N=25 and 30 revolutions led to the formation of vortex structures as a result of laminar flow of the composite material. Note that similar observations were earlier made in ref. [10]. In the center of samples wide curved layers of the starting materials are observed (figure 2a). At the mid-radius area of samples, the initial materials were transformed into a dispersed layered structure with a thickness of the observed layers not more than 100 nm (figure 2b). At the periphery of the samples, some mixed structure consisting of separate areas of dispersed interlayers of the components was formed (figure 2c). In this case, the increase of the revolution number during HPT from 25 to 30 expanded significantly the regions of a homogeneous structure while maintaining small individual zones with a layered structure (figure 1). In addition, during HPT at room temperature and N=25 and 30, the aluminum based solid solution supersaturated with niobium was formed, followed by strain-induced ageing leading to synthesis of the dispersed particles of the Al₃Nb intermetallic phase. This conclusion is based on the results of X-ray diffraction analysis and TEM observations (figure 3).
Figure 1. BSE images of polished cross sections of Al-Nb composites obtained using different initial diameters of Al discs and HPT conditions (N and ω). Dark contrast corresponds to pure aluminum and bright contrast corresponds to pure niobium.

Figure 2. The typical BSE images of the the Al-Nb composites produced by HPT at N=30 and ω=2 rpm: in the center (a), in the mid-radius (b), at the periphery (c) of the sample.

Figure 3. X-Ray diffraction pattern obtained for the Al-Nb-Al samples using Al disc diameter of 10 mm, processed by HPT at ω=2 rpm and N=10 (a); N=25 (b); N=30 (c).

Figure 4. TEM bright-field image and the selected area diffraction pattern obtained from the mid-radius area of the Al-Nb-Al sample using Al disc diameter of 10 mm, processed by HPT at N=30 and ω=2 rpm. Some of the Al$_3$Nb intermetallic precipitations are arrowed (a). Selected area diffraction pattern corresponds to the mid-radius area (b).
Figure 5 represents the results of the microhardness measurements of different samples subjected to HPT with $\omega=2$ rpm. So, at $N=10$ in the center and the mid-radius of samples, in which large inclusions and layers of Nb in Al were inhomogeneously disposed, the microhardness was comparable with that of the deformed Al sample and amounted to about 75 HV. At the periphery of the sample, in which dispersed inclusions of Nb in Al were allocated, the microhardness increased and reached about 110 HV.

The increase of the revolution number generally led to the increase of the microhardness. In this case, the changes of the microhardness along the diameter of the samples subjected to HPT at $N=25$ and 30 revolutions were similar. In the center of samples, where the microstructure was refined in the least extent and distinct dispersed layers and inclusions of Nb in Al were observed, the microhardness was minimal and amounted to about 100 HV in the both states (at $N=25$ and 30). The increase of the strain value from the center to the periphery of samples led to the increase of microhardness to the maximum values about 280 and 300 HV (for $N=25$ and $N=30$, respectively) in the mid-radius. This resulted from the laminated structure with enhanced area of interlayers and the higher density of defects. At the periphery of the samples the maximum value of shear deformation resulted in the formation of the strain-induced solid solution, which in turn resulted in the decrease of the interface hardening component, which was probably not compensated for by the solid solution hardening. Therefore, at the periphery of the samples the microhardness decreased to values about 130 HV (at $N=25$ and 30).

![Figure 5. The microhardness measured along the diameter of the cross sections of the Al-Nb-Al composite samples obtained by HPT (with initial Al disc diameter of 10 mm) and the pure Al and Nb discs in the initial state and after HPT.](image)

4. Summary
Monolithic composite samples based on the Al-Nb hybrid system free of any cracks were successfully produced by HPT using different processing conditions. Optimal combination of the initial disc diameters of Al and Nb was experimentally chosen. It was 10 and 12 mm for Al and Nb discs, respectively. HPT of the discs of the chosen diameters provided the most intensive fragmentation and stirring of niobium in the aluminium matrix. It was revealed that the best fragmentation and the most uniform distribution of Nb in the Al matrix were attained at a rotation rate of 2 rpm. The expansion of the significant structural refinement area and the formation of the metal-matrix structure took place at the revolution number of 25 and 30. The formation of the hardening intermetallic Al$_3$Nb phase due to the occurrence of the strain-induced ageing has been proven by means of X-ray diffraction analysis and TEM observations. The microhardness measurements and microstructure observations showed that the changes in the microhardness along the diameter of the composite samples were consistent
with the microstructures formed in the samples after HPT. The microhardness values in the center of the samples were minimum and amounted to about 100 HV, in the mid-radius they were maximum and amounted to about 280 and 300 HV for N=25 and 30, respectively. At the periphery of the samples the microhardness was slightly higher than in the center and amounted to about 130 HV.

Acknowledgments
The present work was accomplished according to the state assignment of IMSP RAS and supported by the Russian Science Foundation (Grant No. 18-12-00440). Experimental studies were carried out using the facilities of the shared services center “Structural and Physical-Mechanical Studies of Materials” at the Institute for Metals Superplasticity Problems of Russian Academy of Sciences.

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