Microstructure and phase composition of Al-Nb metal-matrix composites produced by shear deformation under pressure

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Abstract. Al and Nb billets with a coarse grain structure in the form of thin discs were stacked in the order of Al-Nb-Al and then deformed by shear under pressure. After the deformation the samples were annealed at 500°C for 30 min. The formation of an intermetallic compound Al$_3$Nb was revealed by using X-ray diffraction and energy-dispersive spectroscopy. After post-deformation annealing, the amount of this compound increased. At the same time, the study of microhardness demonstrates a decrease of the value of microhardness after post-deformation annealing.

1. Introduction
The development of new materials is one of the main tasks of materials science, which ensures the progress in the modern technology. As such materials, composites can significantly improve the properties of products for various purposes and therefore play an important role in the development of modern industry.

One of the methods of solid-phase preparation of composites is shear deformation under pressure [1-3]. Such a method of obtaining composite materials combined with post-deformation annealing makes it possible to form a reliable connection of strengthening phase with the matrix.

Recently, the results of studies on the processing of in-situ metal-matrix composites have been reported for such binary systems as Al-Nb [1], Al-Mg [2], and Al-Cu [4, 5]. Of a particular interest is the Al-Nb binary system investigated in [1], where materials with a grain size of less than 0.5 μm were used as billets.

A study on the effect of coarse-grained structure of billets on the microstructure and phase composition of an Al-Nb metal matrix composite obtained by shear deformation under pressure and post-deformation annealing was the aim of this work.

2. Experimental
Al (purity 99.1%) and Nb (purity 99.7%) billets with a coarse-grain structure in the form of thin disks were stacked in the order of Al-Nb-Al in a weight ratio of 22.5% Al to 77.5% Nb. The stacks of thin billets were placed between flat Bridgman anvils and processed under 5 GPa for 5 turns at a rate of 1 rpm at room temperature. Then the obtained samples were subjected to post-deformation annealing at 500°C for 30 minutes.

The phase composition of the samples was studied by X-ray diffraction (XRD) analysis using DRON 4-07 diffractometer with graphite crystal monochromator on the diffracted ray using Cu - Kα radiation.
Energy dispersive spectroscopy (EDS) and microstructure studies were carried out on a VEGA 3 SBH scanning electron microscope with a backscattered electron detector. The microhardness of the samples was determined by Vickers method using an Axiovert 100A optical microscope with an MNT-10 hardness measuring device. The static load applied to the diamond tip for 10 seconds was 100g.

3. Results and discussion

The results of X-ray diffraction analysis of the fabricated composite are shown in figure 1. After deformation, the diffraction maxima of aluminum and niobium and a diffraction maximum from the (002) plane of the intermetallic compound Al₃Nb were found on the diffraction pattern (figure 1a). Post-deformation annealing at 500°C led to the broadening of the maximum at 2θ = 38.4°, which can be associated with the appearance of a diffraction maximum from the (112) plane of the intermetallic compound Al₃Nb (figure 1b). The observed displacements of the diffraction maxima from Al and Nb after post-deformation annealing can be associated with the formation of a solid solution of niobium in aluminum and aluminum in niobium.

Figure 1. X-ray diffraction profiles of the fabricated Al-Nb composite after shear deformation under pressure (a), after post-deformation annealing at 500°C for 30 minutes (b).
Energy dispersive analysis carried out after deformation of a three-layer stack of Al-Nb-Al showed that mixing of niobium and aluminium was not observed in the central region but it occurs at the edge of the sample (figure 2, table 1). Low mixing of the metals in the centre of the fabricated composite is associated with the inhomogeneity of the shear deformation along the radius of the sample.

![Figure 2. BSE image of the Al-Nb composite at a small magnification.](image)

| Point number | Al  | Nb   |
|--------------|-----|------|
| 1            | 100 | 0    |
| 2            | 100 | 0    |
| 3            | 100 | 0    |
| 4            | 100 | 0    |
| 5            | 25,2| 74,8 |
| 6            | 26,2| 73,8 |

Taking into account the X-ray diffraction analysis data (figure 1a), it can be assumed that the formation of the intermetallic phase begins in the process of cold plastic deformation.

The results of microhardness measurements on a fabricated composite after post-deformation annealing and aluminum after deformation for 5 turns are shown in figure 4. The microhardness of the composite is half that of deformed aluminium in the centre area, but more on the edge. It may be due to the formation of intermetallic compounds during deformation and subsequent annealing.
Figure 4. Vickers hardness variation for the Al-Nb composite after post-deformation annealing at 500°C for 30 minutes.

A similar phenomenon was observed in [1], where a similar experiment was carried out, but the initial Al and Nb had an ultrafine-grained structure. The microhardness of the Al-Nb composite after post-deformation annealing at 500°C for 30 minutes decreased [1]. According to X-ray diffraction data, the formation of intermetallic compounds in [1] after post-deformation annealing proceeds more intensively than in this work. In the cases of using billets with ultrafine-grained structure, a high-intensity diffraction maximum of an intermetallic compound is observed in comparison with the case of using blanks with a coarse-grained structure. Therefore, the structure of the starting materials influences the intensity of the phase formation process in the Al-Nb binary system.

This behaviour of microhardness is associated with phase formation processes both upon deformation and upon annealing. This phenomenon requires a further study.

4. Summary
The data of X-ray diffraction, energy dispersive spectroscopy and the results of microhardness measurements suggest that the formation of an intermetallic phase in a stack of thin coarse-grained aluminium and niobium billets, stacked in the order Al-Nb-Al begins during shear deformation under pressure via solid-phase reaction. Post-deformation annealing continues the phase formation process. During shear deformation under pressure of a stack of thin aluminium and niobium billets and post-deformation annealing in a solid solution of aluminium in niobium and niobium in aluminium, inclusions of the intermetallic compound Al₃Nb are formed and grow, i.e. the in-situ metal-matrix composite is formed. The intensity of the phase formation process depends on the structure of the initial billets. The number and rate of intermetallic inclusions Al₃Nb formation increases when using initial billets with an ultrafine-grained structure.

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