Nano-mechanical testing of Al-Nb metal matrix composites consolidated by high pressure torsion after annealing

R R Mulyukov1,2,3, K S Nazarov4, R U Shayakhmetov4, G F Korznikova1, E A Korznikova1,4

1 Institute for Metals Superplasticity Problems RAS, 39 Khalturin St., 450001 Ufa, Russia
2 Bashkir State University, 32 Validi st., Ufa, 450076, Russia
3 Ufa State Petroleum Technological University, 1 Kosmonavtov st., Ufa, 450064, Russia
4 Ufa State Aviation Technical University, 12 K. Marx St., 450008, Ufa, Russia

E-mail: elena.a.korznikova@gmail.com

Abstract. Among different approaches for obtaining composite structures, one can distinguish the approach of severe plastic deformation that allows obtaining bulk structures from strongly dissimilar metals. The application of large strains is usually associated with grain refinement and, in some cases, with the formation of intermetallic phases, both factors contributing to the strength characteristics of the material. This work presents an investigation of nano-mechanical properties of an Al-Nb composite obtained by high pressure torsion and post-deformational annealing. Measurements show that with an increase in the distance from the centre to the edge of the sample, the nanohardness increases from 2.4 to 3 GPa. Young’s modulus varies in the range from 182 to 197 GPa, which indicates the non-homogeneity of the composite sample. The Young’s modulus of the composite after high pressure torsion was found to be higher than that of pristine Al and Nb, which is probably due to the formation of the intermetallic Al3Nb phase, having an increased Young’s modulus according to ab-initio calculations.

1. Introduction
Following the general trend of design of new materials with enhanced characteristics, several approaches of obtaining composites have been developed, among them one can recall friction stir welding processing [1], plasma sintering [2], stir casting [3,4], spray deposition [5] and diffusion bonding [6]. However, all mentioned methods are inevitably associated with a local temperature increase which, in turn can lead to inhomogeneity and degradation of the properties of the final product. In this respect, consolidation using severe plastic deformation by high pressure torsion (HPT) has several advantages comparing to other methods, namely, it allows reaching large strains, avoiding the fracture of the sample, the control of the strain rate allows preventing extra heating, and the flexible size of anvils provides an opportunity to vary the shape and size in the moderate range [7–10]. Up to date, the successful rapid diffusion bonding of Al-Mg [11], Al-Cu[12–14] Al-Ni [15] has been reported, where the exceptionally high hardness of the final product is usually associated with the
strain induced nucleation of intermetallic phases accompanied by a refinement of the structure refinement.

The loading scheme used in HPT devices allows performing the consolidation of powders of different metals, which also results in the formation of metal matrix composites. This approach enables mechanical alloying of strongly dissimilar materials, such as, Al-Ti [16], Al-W [17] and some others, however the purity of the final product in this case is much lower that for those obtained by consolidation of thin discs. A pioneering attempt to perform bonding of two dissimilar metals - Al and Nb by means of disk consolidation was performed by the authors in [18].

The ability of HPT to the synthesis of bulk composites is primarily associated with a significant inflow of strain energy, which is partially accommodated on the formation of new interfaces, and a high concentration of point defects [19], facilitating diffusion in the lattice. Enhanced diffusion, in turn, provides conditions for the mutual solubility of components and the formation of intermetallic phases at temperatures much lower than those in the corresponding equilibrium phase diagram. The phenomenon of point defect assisted mass transfer in nonequilibrium conditions and related phenomena is addressed in [20-23].

The great number of factors affecting the structure and properties of the composite (the initial structure of the material, HPT processing parameters, number of possible intermetallic phases, exc.) require a great diversity of methods of their characterization. Our previous work reported on the microstructural, X-ray and microhardness investigations of the Al-Nb composite obtained by HPT. This work is devoted to the analysis of the nano-hardness and the Young’s modulus evolution of the studied composite performed on the ultrafine level that allows to uncover new features of the structure evolution upon severe plastic deformation and subsequent annealing, which, in turn, can provide a new insight on the possibilities to create composites with tailorable properties.

2. Materials and Methods

Aluminum with a purity of 99.5 wt.% and niobium with a purity of 99 wt.% in the form of disks with a thickness of 0.5 mm and a diameter of 12 mm were used as starting materials for the research. Two disks of aluminum were stacked in the form of a sandwich with one disk of niobium located in the middle. Pressure assisted shear deformation was performed at room temperature on a constrained Bridgeman-type HPT installation with an anvil depth of 0.25 mm at a pressure of 5 GPa with a total number of revolutions N=30. Bulk disk-shaped samples of the Al-Nb mixture with a diameter of 12 mm and 0.5 mm thickness were obtained.

Nanomechanical testing has been performed by means of a basic module of the «NanoScan-3D» equipment, which allows performing a comprehensive study of nanomechanical properties in the load range up to 100 mN by indentation and sclerometry methods, and to study surface topography in the semi-contact scanning probe microscopy mode. We have performed the nanoindentation based on the measurement and analysis of the indentation load-displacement data enabling determination of the Young’s modulus value and nano hardness of the material by analyzing the load-depth curve (see figure 1) with a load of 30 mN, details of the measurement procedure can be found in [24]. The indentation was performed in three areas (R=0, R=R/2 and R=R, see scheme in table 1) on the cross section of the HPT disc sample. Every value given in table 1 is the result of 5 measurements averaging.

Figure 1. General view of the loading scheme (a), loading curve (b) and contact diagram with the designations of the values used in the method of calculating the modulus of elasticity and hardness. Adopted from [24].
3. Results and discussion

Table 1 presents the results of measurements of nanohardness and Young’s modulus in the Al-Nb-Al composite after annealing at 400 and 600 °C. The measurements reveal that after annealing at 400 °C the nanohardness grows with the distance from the center to the edge of the sample, while the Young’s modulus reveals the opposite dependence - one can see a decrease from 218 to 118 GPa when moving from the centre to the edge part of the sample. Analysis of nanomechanical characteristics after annealing at 600 °C revealed an overall decrease in all measured characteristics. Thus, an increase in the annealing temperature resulted in the nanohardness decrease from 3.75 to 3.03 GPa and from 3.26 to 2.44 in the vicinity of the sample edge and the sample center, respectively. From the data presented in table 1, one can also conclude that annealing at 600 °C resulted in the alignment of the Young modulus value along the sample profile, while its overall value was found to be approximately the same as after annealing at 400 °C.

| Zone                | Sample edge (R=R) | Middle radius (R=R/2) | Centre (R=0) |
|---------------------|-------------------|-----------------------|--------------|
| H, GPa              | 3.75              | 2.81                  | 3.26         |
| E, GPa              | 118.5             | 189.75                | 218.31       |

Table 1. The Young's modulus E, and nanohardness H of the composite Al-Nb after annealing

According to experimental estimations in [25], Young's modulus for Nb in different structural states varies in the range 110-160 GPa, which corresponds to our results. The value of Young's modulus of the composite was higher than that of the original components of the composite. This is probably due to the formation of a large proportion of the dispersed intermetallic phase Al$_3$Nb, which, according to first-principle calculations has a value of Young's modulus E=260 [25].

![Figure 2](image2.png)

Figure 2. Nanoindentation imprints and corresponding 3D surface profile obtained on the NanoScan-3D for Al-Nb-Al composite annealed at 400 °C in the center of the sample (R=0) in the middle radius area (R=R/2) and on the edge of the sample (R=R) (see scheme in table 1).

![Figure 3](image3.png)

Figure 3. Nanoindentation imprints and corresponding 3D surface profile obtained on the NanoScan-3D for Al-Nb-Al composite annealed at 600 °C in the center of the sample (R=0) in the middle radius area (R=R/2) and on the edge of the sample (R=R) (see scheme in table 1).
Microstructural investigations of the Al-Nb composite revealed the presence of three main zones along the sample radius, namely a coarse fragment mixture of Al and Nb phases in the center of the sample (at R=0), a fine lamellar type structure at the mid radius (R=R/2) and a supersaturated solid solution in the vicinity of the disc edge (R=R) [18]. Post deformational annealing results in a decrease in the microhardness in R=0 and R=R/2 areas. Meanwhile the annealing induced precipitation of Al$_3$Nb phase contributes to the increase of the sample microhardness in the edge region [18].

The analysis of the nanohardness and Young’s modulus of the annealed samples is in general in good coincidence with the investigations performed in [18]. Thus, in the center of the disc sample, at R=0 for both annealing temperatures 400 and 600 °C (figure 2 a and figure 3 a respectively), one can distinguish separate Al and Nb phases with the size of the imprint in Nb being much smaller than that in Al due to the lower strength of the latter. 2D and 3D images of NanoScan-3D imprints in the cross section of R/2 area reveal a laminated structure (figure 2 b and figure 3 b) that correlates well with transmission electron microscopy studies [18]. This structure is stable with respect to annealing at 400 and 600°C, and, as a result one can see that H and E values, as well as the size of the imprint are fairly stable in the studied temperature interval.

The disc edge region (R=R) is characterized by a relatively homogeneous microstructure with the highest values of H and E. This can be due to the high fraction of Al3Nb phase that constitutes ~12% and ~25% after 30 min of annealing at 400 °C and 600 °C, respectively [18]. Since this estimation has been done for the whole sample, the fraction of the intermetallic phase at the periphery of the sample is expected to be even higher, thus considerably contributing to the local mechanical properties of the composite.

4. Conclusions
The study of the nanomechanical properties of the Al-Nb composite after annealing revealed that an increase in the annealing temperature results in an overall decrease of nanohardness, keeping the gradient of its value along the radius of the sample together with alignment of the Young’s modulus value along the radius of the sample. The increased E value of the composite comparing to the pristine Al and Nb components is explained by the enhanced Young’s modulus of the Al$_3$Nb phase formed during HPT and subsequent annealing.

Acknowledgements
GFK and RRM gratefully acknowledge the support of the Russian Science Foundation, grant No 18-12-00440. The work was partially supported by the State Assignment of Russian Ministry of Science and Higher Education.

References
[1] Mori K, Bay N, Fratini L, Micari F and Tekkaya A E 2013 CIRP Annals 62 673–94
[2] Ujah C O, Popoola A P I, Popoola O M and Aigbodion V S 2019 Int. J. Adv. Manuf. Technol. 101 2275–82
[3] Mistry J M and Gohil P P 2019 Composites Part B 161 190–204
[4] Bihari B and Singh A K 2017 Int. J. Eng. Res. Appl. 7 42–8
[5] Shockley J M, Strauss H W, Chromik R R, Brodusch N, Gauvin R, Irissou E and Legoux J-G 2013 Surf. Coat. Technol. 215 350–6
[6] Kawasaki M, Han J-K, Lee D-H, Jang J and Langdon T G 2018 J. Mater. Res. 33 2700–10
[7] Zhilyaev A and Langdon T 2008 Progr. Mater. Sci. 53 893–979
[8] Kawasaki M, Figueiredo R B and Zhilyaev A P 2020 Adv. Eng. Mater. 22 1901386
[9] Castro M, Pereira P H, Figueiredo R and Langdon T 2019 Lett. Mater. 9 541–5
[10] Mulyukov Kh Ya, Korznikova G F and Nikitin S A 1996 J. Appl. Phys. 79 8584–7
[11] Kawasaki M, Jung S H, Park J-M, Lee J, Jang J and Han J-K 2020 Adv. Eng. Mater. 22 1900483
[12] Korznikova G, Czeppe T, Khalikova G, Gunderov D, Korznikova E, Litynska-Dobrzynska L and Szlezynger M 2020 Mater. Charact. 161 110122
[13] Bachmaier A, Rathmayr G B, Bartosik M, Apel D, Zhang Z and Pippan R 2014 Acta Mater. 69 301–13
[14] Korznikova G, Kabirov R, Nazarov K, Khisamov R, Shakhmetov R, Korznikova E, Khalikova G and Mulyukov R 2020 JOM 846 156380
[15] Edalati K, Toh S, Watanabe M and Horita Z 2012 Scripta Mater. 66 386–9
[16] Sun Y, Aindow M, Hebert R J, Langdon T G and Lavernia E J 2017 J. Mater. Sci. 52 12170–84
[17] Edalati K, Toh S, Iwaoka H and Horita Z 2012 Acta Mater. 60 3885–93
[18] Korznikova G, Korznikova E, Nazarov K, Shakhmetov R, Khisamov R, Khalikova G and Mulyukov R. 2020 Advanced Engineering Materials 2000757
[19] Korznikova E A, Mironov S Yu, Korznikov A V, Zhilyaev A P and Langdon T G 2012 Materials Science and Engineering: A 556 437–45
[20] Korznikova E, Sunagatova I, Bayazitov A, Semenov A and Dmitirev S 2019 Letters on Materials 9 386–90
[21] Dmitriev S V, Korznikova E A and Chetverikov A P 2018 Journal of Experimental and Theoretical Physics 126 347–52
[22] Zakharov P V, Korznikova E A, Dmitriev S V, Ekomasov E G, Zhou K 2019 Surface Science 679 1-5
[23] Barani E, Korznikova E A, Chetverikov A P, Zhou K, Dmitriev SV 2017 Physics Letters, Section A: General, Atomic and Solid State Physics, 381 3553-3557
[24] Anon http://nanoscan.info/eng/, accessed 02.09.2020
[25] Nong Z, Zhu J, Yang X, Cao Y, Lai Z and Liu Y 2012 Physica B: Condensed Matter 407 3555–60