OBTAINING SUBMICROCRYSTALLINE AND NANOCRYSTALLINE METALS AND ALLOYS BY DYNAMIC CHANNEL-ANGULAR PRESSING

I.G. Brodova[1], V.I. Zeldovich[1], I.V. Khomskaya[1], E.V. Shorokhov[2], A.N. Petrova[1], A.E. Kheifets[1], N.Yu. Frolova[1], I.G. Shirinkina[1], D.N. Abdullina[1] , K.V. Gaan[2]

[1] Institute of Metal Physics Ural branch of the Russian Academy of Sciences, Ekaterinburg, Russia
[2] RFNC-VNIITF, Snezhinsk Russia

We present the experimental data on structural phase change regularities in titanium, aluminum, copper and their alloys. The materials are obtained in submicrocrystalline and nanostructural states by dynamic channel-angular pressing (DCAP) technique developed in RFNC-VNIITF (RF Patent 2283717, Report №26 – 64 p. 2006). In the given paper we give consideration to evolution of structure morphological and sizing properties depending on deformation mode. We consider mechanisms of deformation and metal and alloys strain hardening within the wide range of deformation velocities. We present new data on thermal stability of submicrocrystalline materials obtained by dynamic pressing and also on their dynamic properties under shock wave compression. In unified terms we describe principles of making submicrocrystalline and nanocrystalline materials, different in composition and properties, obtained under dynamic impact.

In a most complete form the investigation results are given in monograph "Structure and Properties of Submicrocrystalline Non-Ferrous Metals and Alloys under Extreme Conditions"/ under the editorship of I.G. Brodova. – Ekaterinburg, UMT UPI, 2018, 363 pp. We note that the closest analog of DCAP is the established equal channel angular pressing (ECAP) technique. DCAP technique is based on high-speed deformation in the material under investigation when moving in a metal matrix with channels intersecting at the angle of 90^0 at a speed of some hundreds meters per second. Specimen acceleration is accomplished by powder gases. Magnitude of specimen deformation velocity is up to ~ $10^5 sec^{-1}$. It is six orders greater than at ECAP. It is stated that the material resulting structure at DCAP is under the effect of simple shear high-speed deformation, compression shock-wave deformation and temperature. Here we present some results and regularities of structural phase changes in titanium, aluminum, copper and their alloys obtained in submicrocrystalline and nanocrystalline states by DCAP technique.

Titanium

We consider structural changes under titanium uniform and localized high-speed deformation as well as peculiarities of crack initiation and development at DCAP. Homogeneous deformation at DCAP produces submicrocrystalline structure in titanium (Fig.1). Following the specimen first pass through the channels the heterogeneous structure if formed with elongated grains-subgrains of section size from 0.3 to ~ 1 μm. Following the second pass the elongated grains-subgrains are converted into equiaxial ones and their disordering increases. The grains size is the same as of grains-subgrains section size following the first pass. The forming microstructure following the second pass exhibits greater degree of uniformity.

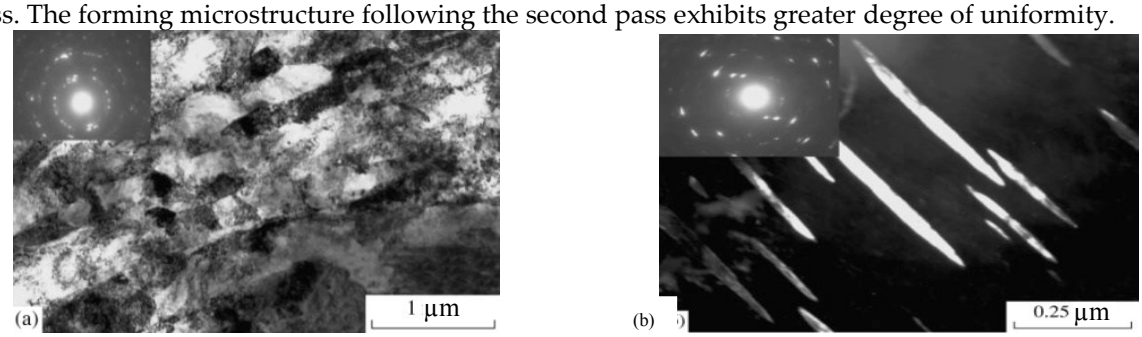

Figure 1. Electron micrographs of titanium structure in the area of homogeneous deformation. (a) – prevailing structure type and twins, (b) - dark-field micrograph

Localized high-speed deformation in titanium results in initiation of adiabatic shear bands (ASB) (Fig. 2 (a)). In specimens loaded according to a single-pass scheme the two band systems are formed: longitudinal bands and vertical bands related angularly to longitudinal ones. Recrystallization inside ASB indicates the material heating up to 770-870K (Fig. 2. (b)). ASB structure at DCAP features their wide width (up to 100 μm) and multilayer structure. Pass number increase up to two is accompanied by accumulation of localized deformation sections.

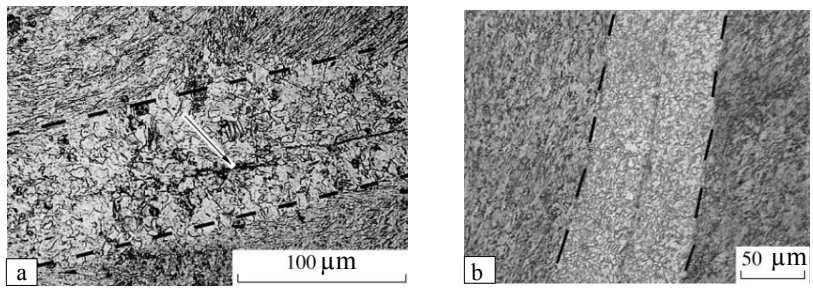

Figure 2. Titanium specimen microstructure in localized deformation sections:(a) - adiabatic shear band along a crack, (b) - recrystallized grains in adiabatic shear band

Temperature increase at titanium dynamic channel-angular pressing prevents the destructing and forming adiabatic shear bands, which occur at pressing under room temperature. Titanium structure exposed to DCAP at elevated temperatures (500°C and 530°C) appears as dispersed mixture of small recrystallized grains (from 1 μm to 3 μm) and deformed nonrecrystallized sections (Fig. 3 a). Following DCAP at 500°C recrystallized grains are arranged into elongated inclined bands and short longitudinal chains. The deformed sections are of sub-grain structure with sub-grains sections 200-300 μm in size. At the second pass the recrystallized grains size is reduced by half (Fig. 3 b), the sub-grains in the deformed sections assume a more equiaxial form, and microstructure becomes more uniform. The obtained duplex structure looks like titanium structure following the rolling deformation at elevated temperatures (150-450°C) and subsequent fractional (short-term) recrystallization at 600°C. However, size of recrystallized grains following DCAP is 3-4 times smaller than following rolling and recrystallization.

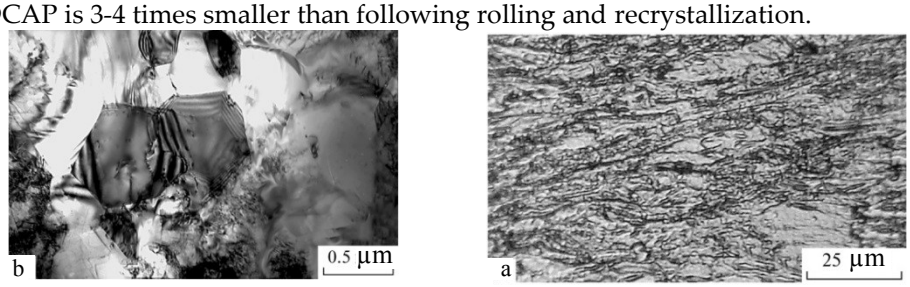

Figure 3. Titanium microstructure following two-pass DCAP at 530°C (a), recrystallized grains (b) transmission electron microscopy

Warm rolling of the specimens exposed to DCAP at elevated temperatures increases dislocation overall density and provides high level inherent stress. Two-pass DCAP for titanium at 530°C permits one to get ultimate strength 650 MPa at percentage of elongation 19%. Additional rolling for 50% at 300°C and annealing at 210°C increases ultimate strength up to 790 MPa on retention of rather high percentage of elongation (Fig. 4).

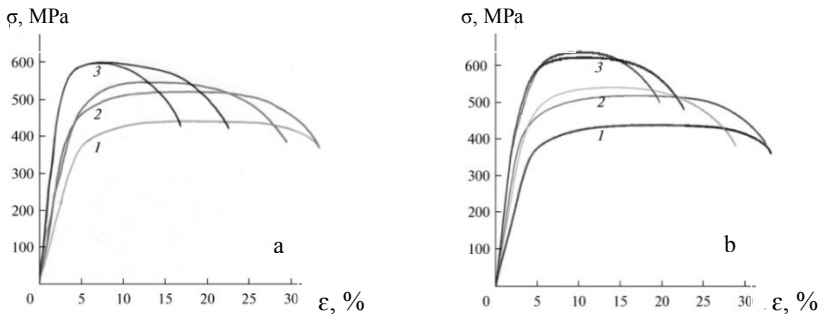

Figure 4. Titanium stress-strain diagram: a – DCAP temperature effect, (b) – effect of pass number. Diagrams: 1 – initial titanium; 2 – DCAP at 500°C; diagrams 3 in (a) – DCAP at 390°C, diagrams 3 in (b) – two-pass DCAP at 530°C

Aluminum alloys

We studied the regularities of generating submicrocrystalline aluminum alloys of various composition by DCAP. We divided alloys into two groups depending on matrix doping level: non-heat-treatable alloys Al-Mg–Mn and Al-Mn alloys with light solid-solution hardening (АМц and A5083) and heat-treatable alloy Al-Zn-Mg-Cu with dispersion and strong solid-solution hardening (B 95). The essential features of fragmented submicrocrystalline structure in Al alloys with strong solid-solution hardening are: fragment size - 200 nm, prevalency of large-angle boundaries dividing crystalline grains (their proportion is 50-60%); diffuse, nonequilibrium boundaries of crystalline grains; nonuniform diffraction contrast inside crystalline grains being evidence of high level inherent stress; lattice dislocations high density ~10^{15} m^{-2} (Fig. 5a).

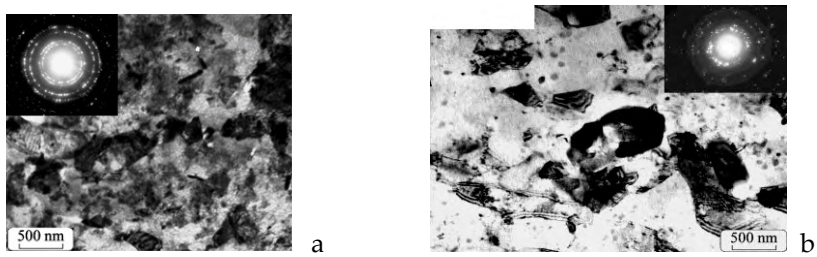

Figure 5. Al alloys structure electron microscope image following DCAP at V=150 m/sec: (a) – alloy B 95, N=2, (b) – alloy АМц, N=4

Cumulative deformation value increase with a rise in a specimen initial velocity at DCAP in non-heat-treatable alloys (АМц, A5083) leads to substitution of the fragmented structure for a mixed one, in which dynamically recrystallized grains occur (Fig. 5b). Submicrocrystalline structure of this type features lower-level elastic stress and less density of dislocations. At equal average grain size the grain size distribution is of bimodal nature, and a fraction of large-angle boundaries increases. The noted peculiarities of structural transformations in various composition alloys make an impact on deformation hardening value, which is higher for the fragmented structure.

We obtained results on mechanical behavior measurements in a wide range of various-grain aluminum alloys deformation velocities — from micron to submicron scale. Relying on evaluation of experimental data on static loading and dynamic behavior determined at compression by Hopkinson-Kolsky scheme, and also at plane shock wave loading we constructed velocity function (from 1,4 10^{-3} sec^{-1} to 7 10^{5} sec^{-1}) of yield point of submicrocrystalline structure alloys АМц and B95 of grain size 200-600 nm. For the investigated submicrocrystalline structure alloys the dependence of yield point on deformation velocity is of complex nature and depends on submycrocrystalline structure type. The inverse velocity dependence of yield point for B95 alloy discovered within the range (4-6) 10^{3} sec^{-1} could be accounted for substitution of dislocation sliding for grain boundary sliding as for deformation mechanism.

When comparing with properties of coarse crystalline alloys of a similar composition it is apparent that at shock wave compression all the strength characteristics of submicrocrystalline structure alloys B95, АМц and A 5083 appear to be higher (Fig. 6). For instance, following DCAP in alloy A5083 the maximum Hugoniot elastic limit σ_{HEL}=0.66 GPa is obtained, that exceeds σ_{HEL} of coarse crystalline alloy by 78%. Dynamic limit of yield point is Y = 0.31 GPa, that exceeds yield point coarse crystalline alloy by 63%.

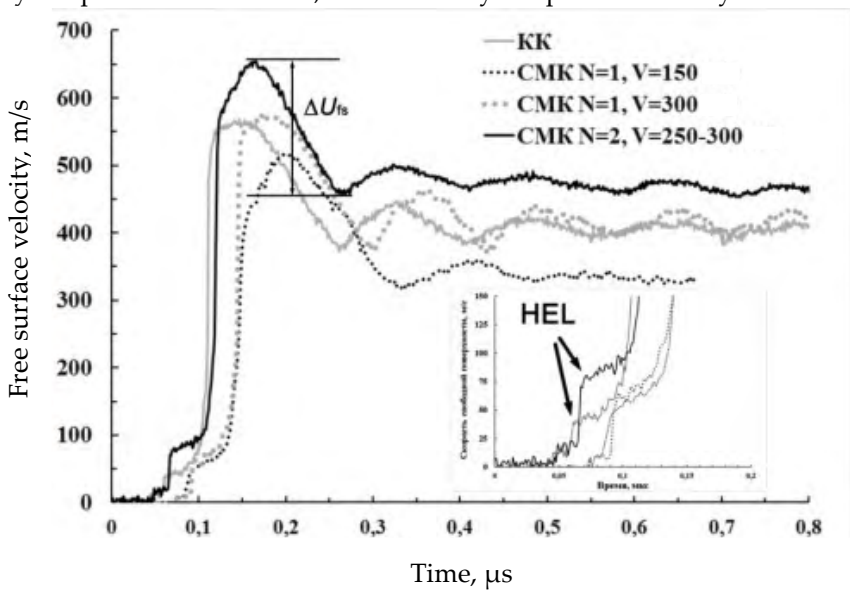

Figure 6. Free surface velocity profiles for submicrocrystalline structure и coarse crystalline specimens of A5083 alloy obtained at shock wave compression by laser-interferometric technique

With X-ray tomography and raster electronic microscopy we investigated process of deterioration of submicrocrystalline structure alloy A5083 following shock wave compression and determined fracture of material of grain size nonhomogeneity (coarse crystalline —40 μm and submicrocrystalline — 400 nm). We carried out the statistical analysis of such defects as pores and micro-cracks. We plotted a porosity curve as a distance function of fracture surface and calculated fracture zones width. The obtained data could be applied to verify materials fracture models at impulse action.

Copper and its alloys

When studying copper structure evolution at high-speed (10^4-10^5 sec^{-1}) severe plastic deformation we stated with DCAP technique the significant, three order from 100 μm to 0,1 μm, copper structure refinement at DCAP. It can be obtained even at one- and four-time pressing of a specimen through channels unlike quasi-static technique ECAP, when submicrocrystalline structure in copper is obtained at eight- or twelve-time pressing of a blank. It is discovered that structure formation in copper is evaluated by regularly recurring high-speed processes of fragmentation, dynamic polygonization and fragmental dynamic recrystallization. It is demonstrated that four-pass DCAP results in formation of uniform nonequilibrium (submicrocrystalline + nanocrystalline) structure made of grains-subgrains 50 nm to 400 nm in sizes, through the whole length of a copper specimen, and number of grains-subgrains 50-100 nm in size amounts up to 35%. (Fig. 6). Copper with (submicrocrystalline + nanocrystalline) structure possess high mechanical characteristics: Hv=1560 MPa, $\sigma_в$ = 440 MPa, $\sigma_{0,2}$ = 414 MPa and δ = 19%. Copper hardness (submicrocrystalline + nanocrystalline) increases 1,8-2 times, strength increases 1,4 times on retention of plasticity satisfactory level as compared with original coarse-grained state.

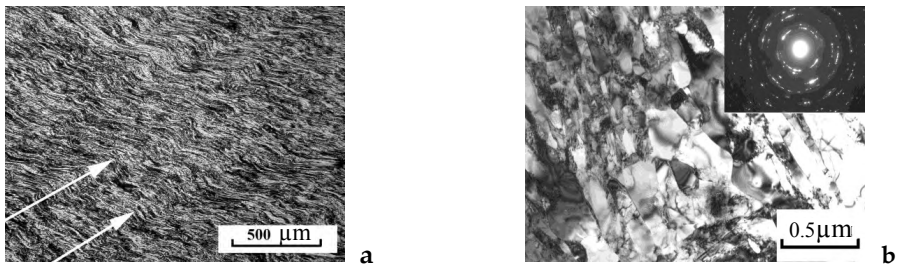

Figure 7. Copper specimen structure following DCAP four passes:
(a) – fiber structure,(b) – grains-subgrains inside copper fibers

It is stated that copper specific electrical resistivity with nonequilibrium submicrocrystalline + nanocrystalline structure obtained at DCAP and measured at cryogenic temperature (4,2 K) significantly (fivefold) exceeds copper specific electrical resistivity in completely annealed coarse-grained state.

We studied evolution of micro- and low alloys structure based on Cu-Zr and Cu-Cr-Zr systems and investigated their properties. It is stated that deformation ageing with release of minor phase nanoparticles (< 5 nm) partially develops in copper micro- and low alloys at DCAP along with processes of fragmentation, dynamic polygonization (Fig. 7a).

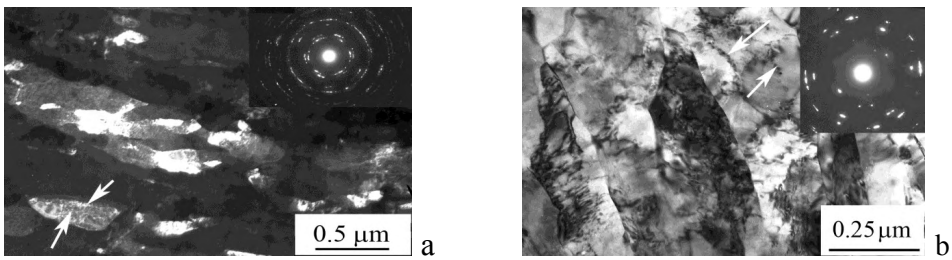

Figure 7. (a) – Submicrocrystalline structure of Cu–Cr–Zr alloys following DCAP, dark-field image in reflex $002_{\alpha'}$, (b) - DCAP + annealing (ageing) at 400°C, bright-field image

Comparative analysis with microdurometry and kinetic microindentation showed that Vickers hardness, Martens hardness and indentation hardness is approximately 1,4 times as much as compared with original coarse crystalline state in copper with submicrocrystalline + nanocrystalline structure. Still more significant increase of the above-noted strength characteristics (2,4; 2,5 and 2,8 times) at simultaneous increase of contact elastic modulus by 21-27% is obtained in alloy Cu-0,09Cr-0,08Zr with submicrocrystalline structure containing strengthening phase nanoparticles.

We investigated physical and mechanical properties of copper alloys with submicrocrystalline structure obtained at DCAP and containing minor phase nanoparticles. It is shown that the investigated Cu-Zr and Cu-Cr-Zr systems alloys with chrome (0,09-0,14%) and zirconium (0,04-0,08%) as microadditives are of submicrocrystalline structure and contain minor phase nanoparticles. They are stated to be thermally stable even at heating up to 500-600°C. Figure 8a shows ageing temperature effect onto micro-hardness of quenched and submicrocrystalline structure alloy Cu-0.14Cr-0.04Zr. Heating submicrocrystalline structure alloys up to 200-300°C does not lead to micro-hardness change. It is observed to increase up to 1700-1780 MPa at 350-450°C due to solid solution decay processes. We note that ageing duration varying at 400°C from 1 to 4 hours increases micro-hardness of submicrocrystalline structure alloy up to 1880 MPa. Heating up to 550-600°C leads to micro-hardness reducing (Fig. 8a, curve 2), that is due to recrystallization process development. We stated the regimes of significant enhancing the mechanical properties of Cu-0.14Cr-0.04Zr alloy with submicrocrystalline structure containing minor phase nanoparticles. Thus, ultimate strength increases 2.8 times, and yield point – 5.1 times as compared with original coarse crystalline state. In doing so, alloys maintain satisfactory plasticity. Electrical resistance of Cu-0.14Cr-0.04Zr alloy with submicrocrystalline structure containing minor phase

nanoparticles obtained at DCAP and ageing at 400°C is 2,1-2,3 microohm·cm, that exceeds electrical resistance of quenched alloy only slightly (Fig. 8b)

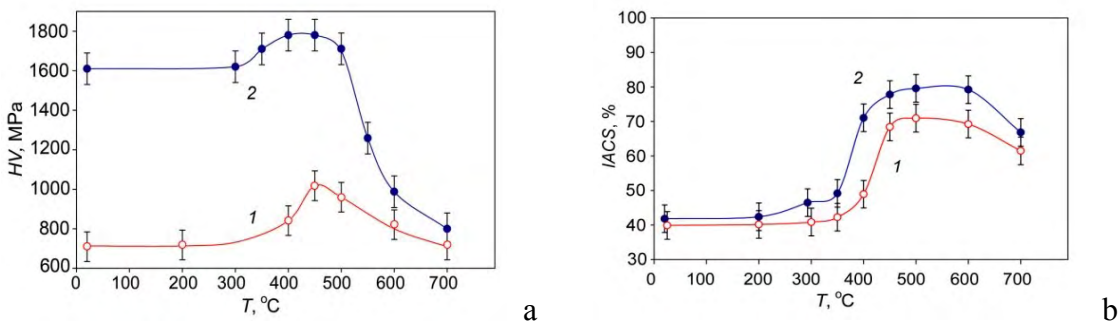

Figure 8. Ageing temperature effect onto micro-hardness (a) and electrical conductivity (b) of alloy Cu-0.14Cr-0.04Zr on quenching from 1000°C- (1) and DCAP - (2)

We conducted investigations on effect of additional quasistatic severe plastic deformation at slipping friction onto evolution of structure and properties of economically-doped precipitation-hardening alloys based on Cu-Cr-Zr system obtained with DCAP. By the example of Cu-0.09Cr-0.08Zr alloy it is stated that wear intensity of specimens with submicrocrystalline structure obtained at DCAP is reduced by 1.4 times as compared with coarse crystalline state. It is stated that combined treatment DCAP+400°C+ severe plastic deformation at friction results in formation of surface nanocrystalline layer with crystallites 15–30 nm in size in the material, that provides high-level hardness (3350 MPa) and satisfactory tribological properties of alloys. The conducted works result in a conclusion about potentials for application of the developed technology as a candidate for obtaining materials with enhanced physical and mechanical properties.

СИСТЕМА ИЗМЕРЕНИЯ ПАРАМЕТРОВ ВОЗДУШНЫХ УДАРНЫХ ВОЛН

А.С. Соколов, Д.А. Кондрашов, С.П. Кожарский, Г.А. Бубнов

Федеральный научно-производственный центр "Алтай", Бийск, Алтайский край, Россия

На предприятии АО «ФНПЦ «Алтай» ведется отработка новых составов и боевых частей. Для оценки эффективности их действия необходимо проводить измерения параметров воздушной ударной волны. Таким образом возникла необходимость разработки информационно-измерительной системы.

По результатам научно-технического поиска было выяснено, что наиболее актуальным методом измерения параметров воздушной ударной волны является тензометрический. На основание этого метода разработана информационно-измерительная система (ИИС) АРМ «ВУВ».

Система регистрирует создаваемое при взрыве давление и позволяет оценить следующие параметры:

- импульс фазы сжатия воздушной ударной волны, J_+;
- избыточное (пиковое) давление на фронте ВУВ в заданных точках регистрации, ΔP_m;
- среднюю скорость распространения фронта ВУВ между двумя точками регистрации по лучу измерения, $V_ф$ [1].

На основании полученных результатов обработки профиля ВУВ определяется тротиловый эквивалент испытываемого ВВ.