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THE TEMPERATURE EFFECT ON SEVERELY DEFORMED ALUMINIUM BY HIGH PRESSURE TORSION

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ABSTRACT

The process of deforming materials started to attract the interest of the researchers after the development of the severe plastic deformation techniques.

A number of aluminium samples were severe deformed by HPT method with different deformation degree. The researches follow to determine SPD effect on this material and temperature influence on fine aluminium structure. The paper present XRD and DSC results.

KEYWORDS: severe plastic deformation, high pressure torsion, XRD, DSC

1. Introduction

Over 20 years ago, Herbert Gleiter presented the first concepts for developing nanocrystalline materials, or ultrafine-grained materials with a grain size under 100 nm, with special properties. After his research the field of nanostrucured materials has developed rapidly in virtue of science interest for this field. He stated that because of containing an extremely large fraction of grain boundaries with special atomic structure, nanomaterials should have unusual properties., for example their mechanical properties present very high strength, toughness, fatigue life and wear resistance [1]

After severe plastic deformation, in the material can appear high internal stresses caused by high density of dislocations inside the grains and their boundaries. The presence of non-equilibrium grain boundaries containing numerous grain-boundary dislocations is an immediate consequence of severe straining, but it can be controlled by subsequent annealing or special thermomechanical treatments, or both. It is well known that grain refinement promotes mechanical strength, and thus one can expect ultrafine-grained materials to possess very high strength.

Moreover, introduction of a high density of dislocations in SPD-processed nanometals may result in even greater hardening.

However, all this normally decreases ductility. Strength and ductility are the key mechanical properties of any material, but they are typically opposing characteristics. Materials may be strong or ductile, but rarely both at once. Recent studies have shown that material nanostructuring may lead to a unique combination of exceptionally high strength and ductility (Fig. 1), but this task calls for original approaches[2].

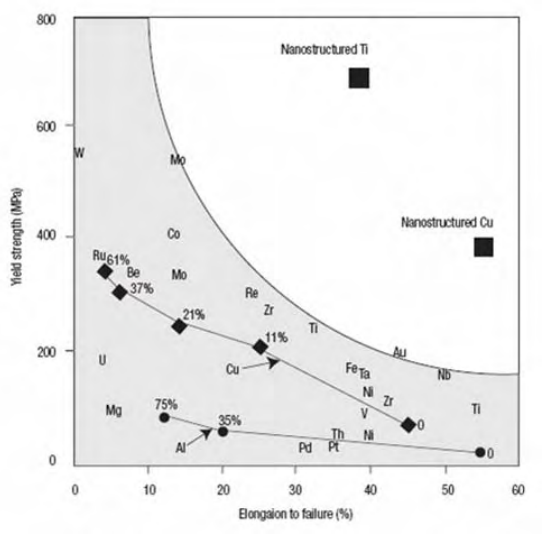

***Fig. 1.** Strength and ductility of the nanostructured metals compared with coarse-grained metals [2]*

Conventional cold rolling of copper and aluminium increases their yield strength but decreases their ductility. The two lines represent this tendency for Cu and Al and the % markings indicate a percentage on rolling. In contrast, the extraordinarily

high strength and ductility of nanostructured Cu and Ti clearly set them apart from coarse grained metals[2]. Experiments have shown that in SPD produced metals, the diffusion coefficient grows considerably (by two or three orders), and this is associated with non-equilibrium grain boundaries. So perhaps grain-boundary sliding is easier in these ultrafine-grained metals and develops during straining even at lower temperatures, producing increased ductility. It is well known that enhanced sliding in nanostructured metals can lead even to superplasticity at relatively low temperatures[2]. Methods of severe plastic deformation can provide formation of nanostructures in different materials. However, an obtained grain size and a character of a nanostructure forming depend on the SPD methods applied, processing regimes, phase composition and initial microstructure of a material. Below, are examples of typical nanostructures.

2. Experimental procedures

The samples cylinder shape were severe deformed in lab using HPT constraint scheme. The rotating movement is applied on the upper punch. Also the upper punch has no vertical movement in SPD process. The necessary force is applied on the inferior punch using a hydraulic system. The samples having cylindrical shape were deformed in a number of steps (table1). The force variation is registered with an accurate Spider 8 Hottinger system.

***Table1**. SPD parameters and samples dimensions*

Sample Φ=9.4mm The starting material for Al suffered repression		**Height**		**Force**	**Rpm**	**Nr of SPD pass**	**Experiments**	
Name	**No.**	**Ho**	**H1**				**DSC**	**X-Ray**
		[mm]		[N]	[N/mm^2]			
I Al	1	6.4	5.4	2016.968	180	1		
	2.1	4.8	2.9	15604.96	180	2		
	3.2	2.9	1.9	28280.01	180	3		
	4.3	1.8	1.4	59744.71	180	4		
	5.4	*1.2*	*0.7*	*54946.45*	*180*	*5*	*X*	*X*
II Al	1	4.4	1.4	42059.09	180	1		
	2.1	1.5	1.2	39744.88	180	2		
	3.2	1.1	1.0	34628.15	180	3		
III Al	1	4.4	1.9	19023.19	180	1		
	2	4.5	1.6	19511.51	180	2		
	3	4.4	1.3	19214.27	180			

2.1. Methods of characterisation

X-ray structural analysis is a method providing important data on the defect structure of NSM (nanostructured materials). X-ray patterns of nanostructured materials processed by SPD methods differ significantly from X-ray patterns of corresponding coarse-grained materials.

These differences are revealed, first of all, in changes of integral intensity of background, changes of width and intensity of X-ray peaks and appearance of crystallographic texture. The background on X-ray patterns is a result of diffuse scattering of X-rays.

The recent results of calculations show that the integral intensities of the background on the X-ray pattern of nanostructured material exceed the corresponding value for coarse-grained material. The increase in the integral intensity of the background indicates an elevated density of crystal structure defects and a possible change in the vibrational spectrum of atoms in nanostructured copper. The XRD analyses were made in CENIMAT laboratories Portugal. The equipment currently available in the Laboratory of X-ray diffraction of CENIMAT is a system Rigaku, Dmax III-C model and a system based on a Siemens rotating anode generator.

***Fig. 2.** The XRD Lab in the CENIMAT, Portugal*

2.2. The XRD samples preparation

The Al sample was studied on X-Rays and DSC. First it was done the DSC analysis to establish the temperature cycle for the XRD analysis.

Before preparing the sample, it was taking in account what surface of the sample shell be studied, so it was chosen the surface that is more sharp than the other. After it was taking in account the geometry of the sample, and it was cut where the surface was symmetrical and parallel to the sample-holder after had been arranged in the cavity.

The preparation for the XRD analysis starts by cutting a rectangular piece from the deformated sample to mach the sample-holder's cavity (L =14mm, l =10mm, H= 0.2; 0.9).

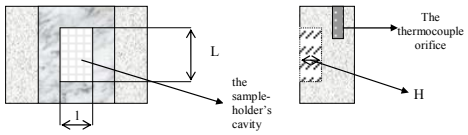

***Fig.3.** The sample-holder*

It is very important to know that the deformed samples can't be polish because we risk loosing the information that will help us analyse the surface of the sample.

The NITI samples where cleaned in acid from the oxides on the surface and after their where clean with alcohol. The rectangular piece was glued (the glue that we used is Araldit rapid with a little bit of silver to make it stronger) in the cavity of the sample-holder and placed inside the cavity vertically therefore to be radiated with ka beam. After the thermocouple is introduced in a special hole inside the upper sample-holder and it will measure the temperatures that it will reach during the temperature cycle.

After is checked that the sample-holder is well introduced in the special place and the sample doesn't fall out of the cavity, the installation is prepared for the analysis by checking that the doors are well closed, setting the intensity to be 100-150 mA and the and voltage 30-35 KV, vacuuming the enclosure where the sample is staying.

The alignment of the beam and sample holder can start. For the alignment we used a limitation for the X Ray with an aperture of 1 mm and a copperplate. After the alignment the copperplate was change with a plate with a different aperture of 0.6mm it is necessary for the alignment to be done because it has to be corrected by rotation and translation the way of the beam above the surface of the sample.

After the alignment is done and the parameters are well established, the analysis can start on a different cycle of temperatures for each different sample. A full scan lasted for about 5-7 hours, for each sample. The results where saved on different files for each temperature. With the peaks that where shown in the scans there were build in Eva program 3D graph and with them it can be seen very well the movement of the peaks and the phases appearing and disappearing. The reading files for all the temperatures from each cycle were saved in different.

After, it was created an EVA file, it can be seen all the transformation at different temperatures. With EVA file we can compare all the scans at different temperatures.

The results were processed in programs like: Microsoft XLS, Origin.

3. Results and discussions

The results for the sample of Al are presented in the previous graphics.

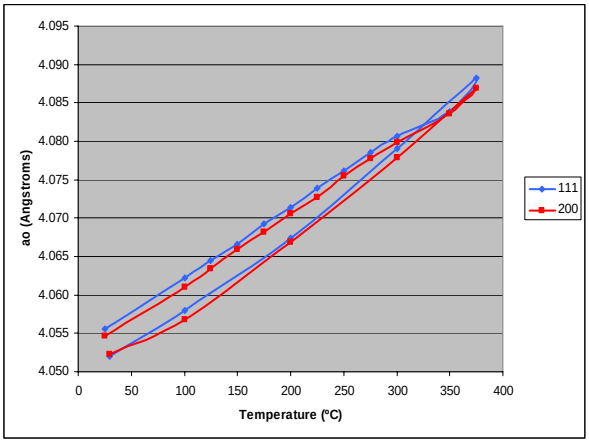

***Fig.4.** The lattice parameter - Temperature*

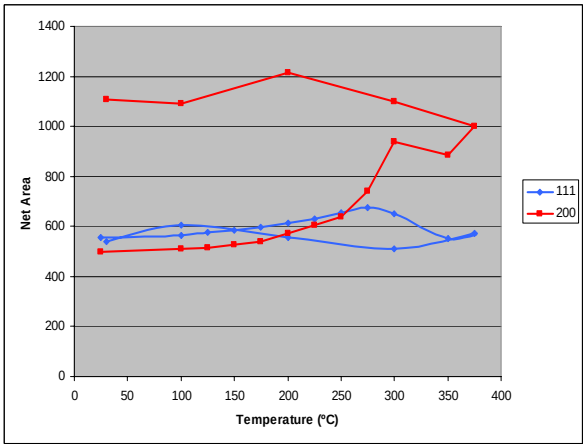

***Fig.5.** Net area - Temperature*

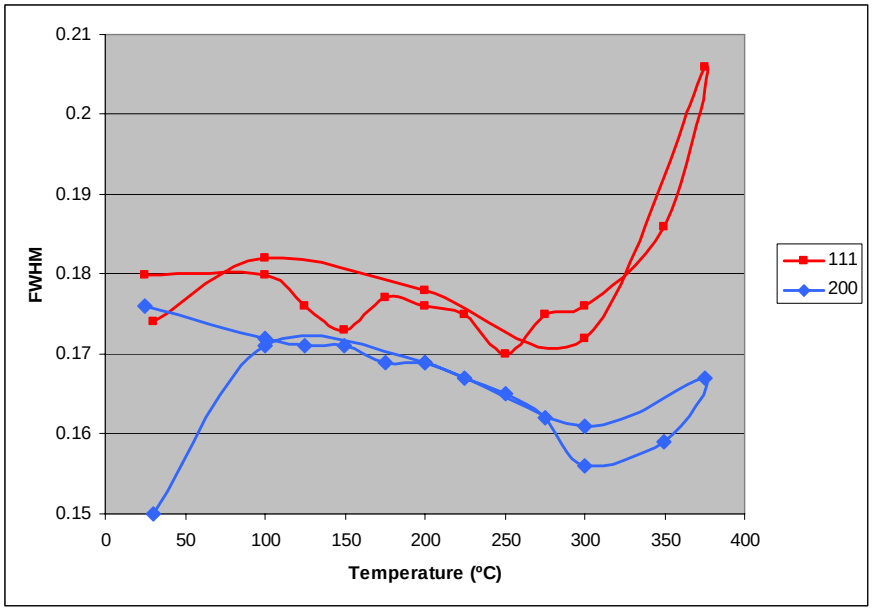

Fig.6. *FWHM (full width at half maximum)-temperature variation*

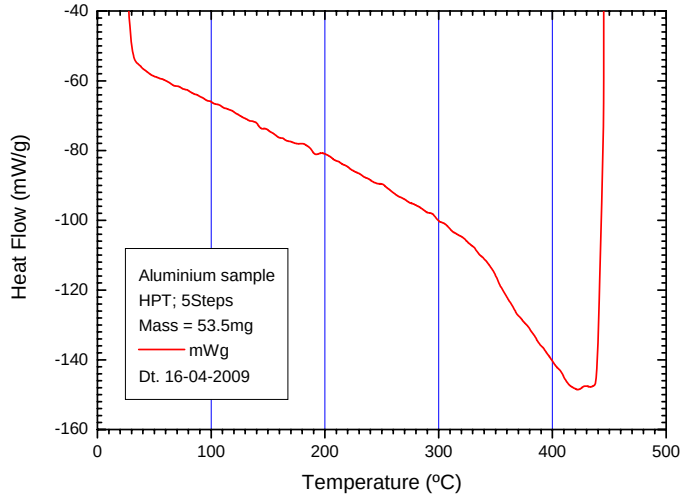

Fig.7. *Aluminium DSC variation*

Looking at the DSC graph, it results that when the sample receives heat, when it reaches the point of 400ºC, in its structure occurs the recrystalisation.

4. Conclusions

In laboratories of University *Dunarea de Jos* of Galati vas developed a SPD technique based on High Pressure Torsion method. Aluminium disk shape samples vas made at different deformations degree. The fine structure vas confirmed after lab tests which consists in X ray diffraction tests and DSC tests. The X ray diffraction tests developed at different temperatures point out behaviour fine aluminium structure.

References

[1]. Ruslan Valiev - *Nanostructuring of metals by severe plastic deformation for advanced properties, article in progress*, Institute of Physics of Advanced Materials,Ufa State Aviation Technical University,12 K.Marx str., Ufa 450000, Russia, p.511

[2]. R.Z. Valiev, R.K. Islamgaliev, I.V. Alexandrov - *Bulk nanostructured materials from severe plastic deformation* , Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, 12 K. Marx Street, 450000 Ufa, Russian Federation, p.51-514

[3]. H.P. Karnthaler, T. Waitz, C. Rentenberger, B. Mingler - *TEM of nanostructured metals and alloys,* Materials Science and Engineering A 387–389, 2004, 777–782

[4]. L. Kommel, I. Hussainova, O. Volobueva - *Microstructure and properties development of copper during severe plastic deformation*, Materials and Design, 2006

[22]. E. Schafler, M.B. Kerber - *Microstructural investigation of the annealing behaviour of high-pressure torsion (HPT) deformed copper*, Materials Science and Engineering, 2006