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Nanoquasicrystalline Al-Fe-Cr-alloy Performed by Quasihydrostatic Compression

Structural characterization and mechanical properties

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***Abstract*—The present research is aimed at the nanoquasicrystalline Al-Fe-Cr alloy consolidated under conditions of quasihydrostatic compression. Water atomized powder of nominal composition $Al_{94}Fe_3Cr_3$ and fraction volume of nano-sized quasicrystalline particles of i-phase about 30% has been used in experiments. Prior quasihydrostatic compression the powder was sieved to the size ranged from 40 to 60 µm. Structural stability of consolidated Al-Fe-Cr alloy in response to heat treatment has been examined by precise method of differential scanning calorimetry and X-ray analysis. The results obtained for consolidated Al-Fe-Cr alloy have been specified and compared with those of the feedstock powder. Mechanical parameters of as-receive and heat treated consolidated Al-Fe-Cr alloy have been determined by using indentation technique and discussed by considering specific features of structure. Nanoquasicrystalline Al-Fe-Cr alloy subjected to quasihydrostatic compression showed high strength and sufficient ductility, maintaining them at the unchanged level up to 400 °C.**

Keywords* — *nanoquasicrystals; Al alloy; quasihydrostatic compression; thermal stability; mechanical properties

I. INTRODUCTION

Quasicrystalline Al–Fe-based alloys, which relate to a new class Complex Metallic Alloys (CMAs) [1,2], is presently of growing attention for researches employed both in scientific and engineering fields of activities. Quasicrystals exhibit a remarkable combination of properties including: high hardness, strength and elastic moduli; low surface energies and frictional coefficients; and good resistance to wear and corrosion [3-7] although they are generally too brittle to be used as a single material. That is why Al–Fe-based alloys comprising a number of quasicrystalline particles dispersed over α-Al matrixes are thought to be the most attractive for potential engineering application. In particular, excellent balance between a high strength and sufficient ductility is indicative of quasicrystalline Al–Fe–Cr-based alloys [8-10]. As to mass production, powder atomization is considered to be the most effective technique [10,11] compared to the other processes available for creation quasicrystalline structure in Al-Fe–based alloys by using required high cooling rate about 10^5 K/s. However, there are significant challenges have to be overcome when producing bulk-shaped materials from quasicrystalline semi-product. State-of-the art, hot extrusion and cold spraying technique as efficient alternative process have been developed to consolidate quasicrystalline semi-products in bulk-shaped composite material [8,10,12-14]. This paper highlights applicability of quasihydrostatic compression for performance of bulk-shaped material based on powdered Al–Fe–Cr-based alloy.

II. EXPEROMENTAL

A. *Materials and Processing*

Powder of quasicrystalline alloy with nominal composition $Al_{94}Fe_3Cr_3$ was used in experiments. Quasicrystalline powder of Al-based alloy with oxygen content about 0.2% was fabricated by water-atomization technique using inhibited high-pressure water with Ph 3.5 [10]. The latter technique provided for fraction volume of nano-sized quasicrystalline particles not higher than 30%. After atomization the powder particles were sieved to the size ranged from 40 to 60 µm using the corresponding sieves. Consolidation of quasicrystalline powder was realized under conditions of quasihydrostatic compression by using high-pressure cell in form of “toroid”. Quasihydrostatic compression was performed at room temperature and pressure of 2.5 GPa.

B. *Structural Characterisation*

Structural characterisation was performed by X-ray diffraction (XRD) analysis using Cu K_α radiation. The icosahedral quasicrystalline i-phase was indexed using Cahn’s indexation scheme [15]. As to determination of lattice parameter for α-Al solid solution, powder of Si as reference material was used in experiments. Scanning electron microscopy (SEM) performed by electron microscope PEMMA-101A (Sumy, Ukraine) was used to study

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microstructural features of baulk-shape material including morphology (size and shape) of quasicrystalline particles. Differential scanning calorimetry (DSC) was employed to study structural stability of feedstock powder and bulk-shaped materials under elevated temperatures. DSC measurements were performed by using STA449F1 analyzer (Netzsch, Germany). Each kind of the sample was placed into DSC cell and exposed under argon flow rate about 20 ml/min during continuous heating from room temperature up to 620 °C. Heating rate used in DSC measurements was as great as 2 K/min. Structural stability of materials was specified by using the samples heat treated during 20 min at the given temperatures.

C. Mechanical Testing

Microhardness measurements were performed using a conventional microhardness machine equipped by standard Vickers’ pyramid. Microhardness numbers were determined under indentation loads not higher than 1.0 N. Yield strength, σ_y, was extracted from ‘stress-strain’ curves constructed by a set of trihedral pyramids with different angles at the tip γ_1 (ranged from 45° to 85°), all adjustable to microhardness machine [16]. Plasticity characteristic δ_H as dimensionless parameter that can generally vary in the range from 0 (for “pure” elastic contact) to 1 (for “pure” plastic contact) was derived by calculations through microhardness, HV, and Young’s modulus, E [17]. Load-displacement measurements were fulfilled to determine Young’s modulus, E, according to the test method procedure proposed by Oliver and Pharr [18].

III. RESULTS AND DISCUSSION

A. Structural characterisation of compressed alloy

The results of XRD analysis for Al-Fe-Cr-alloy subjected to quasihydrostatic compression identify the presence of quasicryatalline icosahedral phase (i-phase) besides α-Al, as can be seen in Fig. 1. Compared to feedstock powder, dislocation density of α-Al solid solution for compressed material was found to be higher by roughly about 3 times while lattice parameter remains the same, as can be seen in Table 1. On the whole, phase transformation of compressed material is very similar to that published previously for atomized powder [19]. As it can be seen in Fig. 1, quasicrystalline i-phase survives in microstructure of the both products at heating up to 400 °C while reflections corresponded to more stable crystalline Al_6Fe-phase become visible in XRD patterns after heat treatment at the temperatures ranged from 450 to 550 °C. Heat treatment temperature of 550 °C and higher favour appearance in XRD patterns of the both products reflections related to stable intermetallic compounds such as θ - $Al_{13}Fe_4$ and θ - $Al_{13}Cr_2$. Attention is paid to the shifting of X-ray peaks for α-Al occurred in response of heat treatment. This fact indicates variation of lattice parameter and, hence, elementary composition of α-Al solid solution. However, phase transformation at heating of compressed alloy was found to be somewhat different compare to that of atomized powder, as evidenced from Fig. 2.

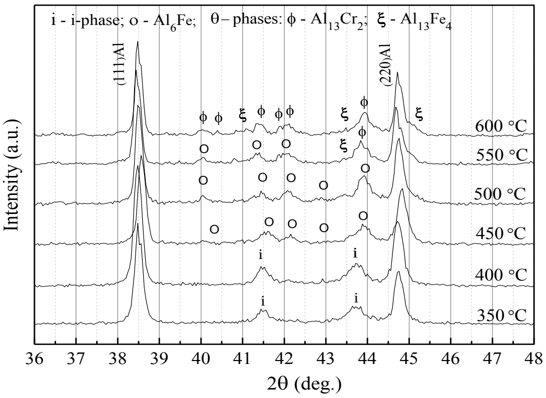

Fig.1. XRD patterns of compressed alloy heat treated during 20 min at different temperatures.

TABLE I. STRUCTURAL PARAMETERS FOR α-Al SOLID SOLUTION

Material	Parameter	
	Lattice parameter, a_o (nm)	***Dislocation density, ρ (m/m^3)***
Atomized powder	0,4047	$2.7\cdot10^{14}$
Compressed material	0,4047	$8.6\cdot10^{14}$

In compressed alloy, lattice parameter of α-Al solid solution is varied markedly. As-opposed to this lattice parameter of atomized powder increases gradually over the all range of heat treatment temperatures, suggesting depletion of α-Al matrix by dissolved Cr and Fe, which atomic radii are smaller by ~ 12% than the atomic radius of Al. It is noticeable that lattice parameter for α-Al solid solution of atomized powder increases significantly as the heat treatment temperature rise up within the range from 350 to 450 °C. The reason of this could be attributed to considerable coarsening of quasicrystalline particles prior they commence to dissolve, resulting in formation more stable Al_6Fe-phase [20]. As to compressed alloy, the difference is that the lattice parameter of α-Al solid solution increases significantly after heat treatment at the temperature lesser by 50 °C than that of atomized powder. However, lattice parameter of α-Al solid solution of compressed alloy falls sharply down to extremely small value after annealing at the temperature of 450 °C, suggesting tremendous enrichment of α-Al matrix by dissolved Cr and Fe. It is noticeable that the latter temperature corresponds to that at which lattice parameter of α-Al solid solution for atomized powder demonstrates rather high value. Distinctive features of phase transformation detected for compressed alloy in response to heat treatment could be explained by considering the imperfection of α-Al matrix and, particularly, the increased content of structural vacancies resulted from nonconservative motion of dislocations. It is presently of common knowledge that the latter process extremely activated under high pressure typical for quasihydrostatic compression. If so, enhanced dislocation density and increased content of structural vacancies can facilitate drastically diffusion of alloyed elements in α-Al matrix, resulting in increased kinetic of phase transformation including coarsening/dissolution of quasicrystals.

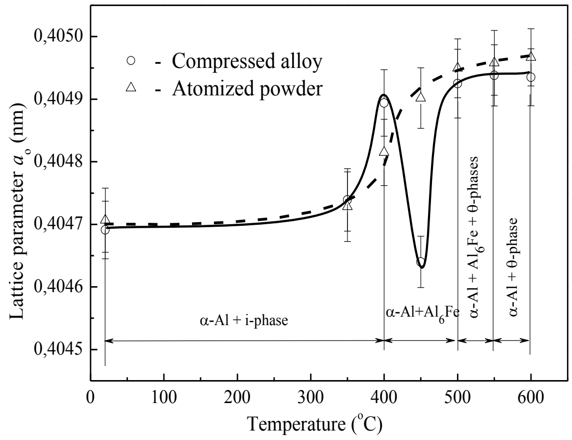

Fig.2. Lattice parameter of α-Al solid solution vs. temperature of heat treatment used for atomized powder and compressed alloy

B. *Structural stability of quasicrystalline Al-Fe-Cr- alloy*

Several exothermic peaks are clearly recognized in DSC runs of the feedstock powder and compressed alloy, as can be seen in Fig.3. Among them exothermic peaks A_1 and A_2 arise from dislocation activity and recrystallization process within α-Al matrix whereas main exothermic peak B is commonly attributed to phase transformation including dissolution of quasicrystalline particles together with simultaneous formation of more stable crystalline Al_6Fe particles and stable intermetallic compounds such as θ-$Al_{13}Fe_4$ and θ-$Al_{13}Cr_2$ [19,20]. Attention is paid to the fact that recrystallization process within α-Al matrix for both kinds of products is going in two-stage, resulting in two exothermic peaks (A_1 and A_2). This kind of process is typical for intermediate and heavy deformed metals/alloys with a stable cellular structure, which induce retardation of conventional recrystallization. As opposed to this recrystallization “*in situ*” when significantly disordered cells act as recrystallization centres becomes more realistic. Actually, high dislocation density for both kinds of products is weighty argument to expect formation of stable cellular structure of α-Al matrix during polygonization at heating. This is especially true for Al-based alloys which have high energy of stacking fault. Moreover, expectancy of stabilizing polygonization for Al-Fe-Cr-alloy is mainly enhanced by the presence in α-Al matrix a number of point defects, i.e. dissolved foreign atoms, precipitate-type defects including nano-sized quasicrystals, and, particularly, excessive vacancies. If that is the case, recrystallization process is controlled by two different mechanisms ascribed to slow and rapid stages [21].

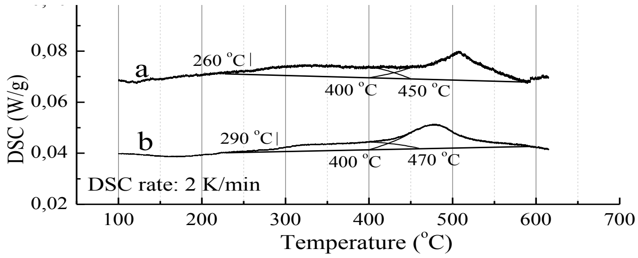

Fig.3. DSC runs for (a) atomized powder and (b) compressed alloy

Slow stage involves very slight growth of cells as temperature increase. Precipitates located lengthways sub-boundaries hinder their migration, preventing cell growth. Rapid stage follows slow one as temperature proceeds to rise up, providing sub-boundaries break away precipitates. It is noticeable that the recrystallization process including slow and rapid stages observed for compressed material shift toward somewhat higher temperatures compared to that of atomized powder, suggesting greater disorientation of cell sub-boundaries due to higher dislocation density. Of importance is the fact concerning superimposition of recrystallization process on the stage of phase transformation, making dissolution of quasicrystalline particles easier. This evidence confirms the assumption derived from the data of XRD analysis.

C. *Mechanical response of compressed Al-Fe-Cr-based alloy*

Fig. 4 shows mechanical parameters such as microhardness, HV, yield strength, σ_y, and plasticity characteristic $\delta_{H,}$ all plotted against heat treatment temperatures. In addition, structural regions marked out in Fig. 2 are also shown in Fig. 4. It can be seen that all of the mechanical parameters keep almost constant values as heat treatment temperature increases up to 400 °C until quasicrystalline particles will be presented in α-Al matrix. Whereupon, plasticity characteristic δ_H of compressed material is found to be just below critical value, $\delta_H = 0.9$, which is presently considered as criterion of ductile behavior of metals and alloys in conventional tests by tensile and bending [17]. However, the values of strength parameters such as microhardness, HV, and yield strength, σ_y, decrease radically as heat treatment temperature increases up to 600 °C when quasicrystalline particles will be dissolved with simultaneous formation **of** stable crystalline Al_6Fe particles and stable intermetallic compounds such as θ-$Al_{13}Fe_4$ and θ-$Al_{13}Cr_2$. On the contrary, the values of plasticity characteristic δ_H increases gradually within the above range of temperature. Generally, mechanical behaviour of compressed material in response to heat treatment is very similar to that of Al-Fe-Cr-based alloy fabricated by the other processing routes [19]. Thus, quasihydrostatic compression is thought to be also potentially appropriate for performance bulk-shaped material based on Al-Fe-Cr alloy which demonstrates excellent balance between a high strength and sufficient ductility.

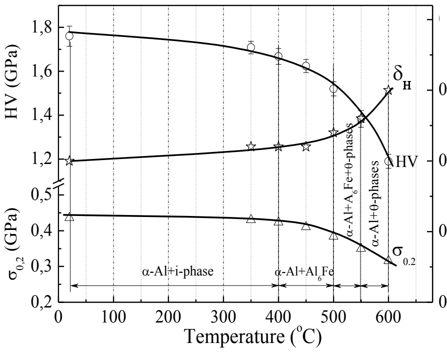

Fig.4. The data for mechanical parameters plotted against temperature used for heat treatment of compressed Al-Fe-Cr-alloy

However, as to mass production, quasihydrostatic compression of Al-Fe-Cr alloy is thought to be more applicable technique compared to hot extrusion currently employed in engineering practice. Moreover, Al-Fe-Cr alloy subjected to quasihydrostatic compression demonstrates higher strength and ductility compared to those indicative of extruded material. Generally, mechanical properties and structural stability in response to heat treatment of compressed material are comparable with those of cold-sprayed alloy [19].

IV. CONCLUSIONS

Structural stability in response to heat treatment of nanoquasicrystalline Al-Fe-Cr alloy subjected to quasihydrostatic compression were found and specified by comparison with that for feedstock atomized powder. Mechanical parameters such as microhardness, HV, yield strength, σ_y, and plasticity characteristic δ_H were measured by indentation technique using contemporary test method procedures.

Evolution of structure for compressed alloy was generally very similar to that of atomized powder. Quasicrystalline i-phase was found to remain in microstructure of compressed alloy at heating up to 400 °C while reflections corresponded to more stable crystalline Al_6Fe-phase become visible in XRD patterns after heat treatment at the temperatures ranged from 450 to 550 °C. Heat treatment temperature of 550 °C and higher favour appearance in XRD patterns reflections attributed to conventional intermetallic compounds such as θ-$Al_{13}Fe_4$ and θ-$Al_{13}Cr_2$.

Dislocation density of α-Al solid solution in compressed alloy was found to be about 8.6 10^{14} m/m^3, exceeding to that for feedstock powder by roughly about 3 times although lattice parameter remains the same. High dislocation density in heavily deformed structure of both kinds of products results in two-stage recrystallization process for which slow stage is followed by rapid one at higher heat treatment temperature. Rapid stage of recrystallization accompanied by dislocation activity superimpose on phase transformation, facilitating dissolution of quasicrystalline phase at heating.

Distinctive features of heat treatment response for compressed alloy consisted in significantly increased kinetic of phase transformation including coarsening/dissolution of quasicrystals at heating. The origin of the matter at issue above was thought to be originated by a number of excessive vacancies resulted from nonconservative motion of dislocation typical for quasihydrostatic compression at high pressure.

Quasihydrostatic compression of Al-Fe-Cr alloy was believed to be more appropriate technique as to mass production compared to hot extrusion currently employed in engineering practice. In addition, structural stability as well as strength and ductility of compressed alloy are higher compared to those indicative of extruded material while they are very similar those of cold-sprayed alloy.

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