Texture of extruded Al-Al_4C_3 material

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Abstract

Dispersion strengthened Al-Al_4C_3 material is characterized by high strength properties and good temperature stability of structure. The wrought products have mostly preferred orientation. The pure aluminium is highly strainable and it induces a particularized texture, common to the material and to the used forming process. This work is focused on a study of the texture in extruded Al-Al_4C_3 materials containing 4 and 12 vol.% Al_4C_3 prepared by powder metallurgy. The aim is an analysis of the effect of Al_4C_3 amount on the texture formation during extrusion and analysis of texture evolution at temperature range from 298 K to 773 K and after cooling to ambient temperature. The extrusion process has induced a very low intensity aluminium deformation texture in the Al-12vol.%Al_4C_3 alloy, but a strong aluminium deformation texture in the Al-4vol.%Al_4C_3 material. The high volume of dispersoid particles consequently efficiently hinders the texture formation during mechanical work. The textures after deformation are in both cases stable up to 773 K.

Key words: dispersion strengthened aluminium, dispersoid, texture, thermal stability

1. Introduction

Aluminium powder metallurgy parts are used for their improved material characteristics or in some cases (e.g. complex shapes) because of their lower production cost. In most of the applications the powder metallurgy parts are used because of one (or more) of the following properties: higher Young's modulus, lower density, higher room temperature strength, better high temperature strength, creep resistance and better wear resistance.

Conventional high strength aluminium alloys such as AA 2618, AA 2219 or AA 2024 loose their strength at temperatures about 473 K, mainly because of rapid coarsening of their precipitates. For certain applications such as jet engine compressors, automotive gas turbines and combustion engine components, aluminium alloys, which retain their strength in the range of 573 K to 723 K, could replace steels or titanium alloys. In the past, several investigations have been made on the formation of recrystallization textures in 6000 series Al-Mg-Si alloys used for car body panels [1, 2].

Dispersions of non-soluble equiaxed ceramic particles in a metallic matrix can enhance the strength and heat resistance of materials (dispersion strengthened material). Ceramic reinforcements are generally oxides, carbides, borides or nitrides (Al_2O_3, Y_2O_3, TiC, TiB_2) [3]. To retain a high strength at elevated temperatures, it is necessary to have a uniform distribution of fine dispersoids, which pin the grain boundaries and harden the matrix. The use of dispersoid phases in aluminium alloys for controlling recovery, recrystallization and grain growth is well established [4].

The first use of aluminium powder for metallurgical application was that by Irmann [5]. His sintered aluminium powder (SAP) is an oxide dispersion strengthened material made from heavily oxidized aluminium flake. It has relatively poor room temperature mechanical properties but good creep strength at temperature above 473 K.

With the advent of mechanical alloying it became possible to put the theoretical concept into practice by incorporating very fine particles in a fairly uniform distribution into often oxidation- and corrosion-resistant metal matrices, e.g. superalloys. Mechani-

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cal alloying is a dry-milling method for processing composite powders with a controlled microstructure. The process was originally developed to produce Ni-based oxide dispersion strengthened superalloys [6]. Progressive materials were prepared by ball milling of atomized Mg powders with various ceramic and carbon nanoparticles that were generated by laser-induced gas phase reaction in a flow reactor [7]. The milling behaviour of ductile materials is characterized by a rapid particle coarsening in the case of aluminium or copper. The introduction of suitable nano-sized dispersoids in the metallic matrix is impossible due to strong welding of the powder particles. To obtain a homogeneous distribution of nano-sized dispersoids in such a ductile matrix a reaction milling is suitable. Reaction milling is a variant of mechanical alloying, which has been established mainly for the production of homogeneous crystalline alloys or alloys with an amorphous structure but also for the manufacture of dispersion strengthened alloys [8, 9]. Suitable dispersoids can be formed by a solid state reaction by introducing components that react with the matrix particular during milling and mainly during the subsequent heat treatment. In the case of Al alloys the introduction of graphite was most successful [10, 11]. The carbon introduced by milling reacts with Al to form fine and well distributed Al_4C_3 particles. Al_4C_3 exhibits high hardness and shear strength, is practically insoluble in Al even close to the melting point and therefore does not tend to coarsen during thermal exposure of the dispersion hardened product. Generally the raw powder mixtures are consolidated by hot extrusion or by hot isostatic pressing obtaining a fully dense material.

Preferred orientation is a very common condition and for alloys it is most evident in wrought products. Particularly, aluminium induces texture during the forming process. In fact, preferred orientation is generally the rule, not the exception, and the preparation of an aggregate with completely random crystal orientation is a difficult task. It is well known that extrusion will cause texture, which influences the properties of extrudate. The pure aluminium is highly strainable and induces a particularized texture, common to the material and to the used forming process [12]. The behaviour of compression deformation and texture formation in Al-Mg alloys at high temperatures has been investigated [13, 14]. Some researchers have used texture coefficient (TC) to evaluate materials texture [15].

In this work it was undertaken a crystallographic orientation study of extruded dispersion strengthened Al-Al_4C_3 material. The aim of this investigation was to try the effect of dispersoid volume on the texture formation during extrusion and to analyse the texture evolution at temperature range from 298 K to 773 K and after cooling to ambient temperature.

2. Experimental

The experimental materials – dispersion strengthened Al-Al_4C_3 materials containing 4 and 12 vol.% Al_4C_3 were prepared by mechanical alloying method. Aluminium powder with particle size below 50 μm was dry milled in an attritor for 90 min with the addition of graphite. The intense milling results in a matrix with fine controlled microstructure and even distribution of carbon particles in the as-milled granulates. The granulates were then cold pressed under pressure of 600 MPa into a cylindrical shape and annealed at 823 K for 30 hours to transform Al and C into Al-Al_4C_3 alloy with desired Al_4C_3 volume fraction. Hot extrusion of cylinders into a bar form was performed at 873 K with 94 % reduction of the cross section.

The samples prepared from the extruded bars (ø 3 mm) in the longitudinal (axial) and transverse (radial) direction with respect to the direction of extrusion were subjected to X-ray diffraction (XRD).

The XRD experiments were performed on the Philips X'Pert Pro diffractometer using the positional sensitive detector-X'Celerator and the high temperature camera-HTK 16 up to 1873 K (Cu Kα radiation, measurement conditions 40 kV/50 mA and divergence slit 0.25 degree). The samples microstructure was so measured directly upon a thermal load. The period of material exposure from 293 K to 773 K was 2 hours and 30 minutes. A qualitative phase analysis was realized from XRD patterns and the texture coefficients were measured. Texture coefficients were calculated from the XRD data using the following equation [16]:

$$\mathrm{TC}_{hkl} = \frac{I_{hkl}/I_{0hkl}}{1/n \sum_{i=1}^{n} (I_{hkl}/I_{0hkl})}, \tag{1}$$

where TC_{hkl} is the texture coefficient for the reflection (hkl), I_{hkl} is the relative diffraction intensity of (hkl) diffraction peak from experimental data, I_{0hkl} is the standard peak intensity for the reflection (hkl) according to JCPDS (Joint Committee for Powder Diffraction Studies) reference file.

3. Results and discussion

3.1. Al-4vol.%Al_4C_3 material

3.1.1. *Measurements at ambient temperature*

Figure 1a documents the XRD lines course in axial direction for extruded experimental Al-4vol.%Al_4C_3 material at ambient temperature and Fig. 1b shows the XRD lines course in radial direction. The qualitative phase analysis of these spectra confirmed the

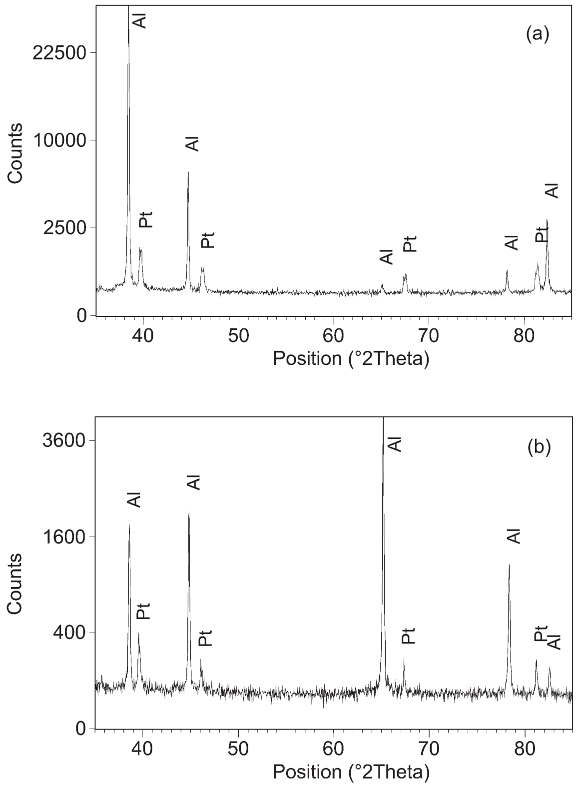

Fig. 1. XRD spectrum of Al-4vol.%Al_4C_3 scanned at ambient temperature and the phase analysis: a) in radial extrusion direction, b) in axial direction.

lines of aluminium and platinum. The presence of the platinum reflections is in consequence of platinum heating body on which is the sample located (by reason of consistent measurements at higher temperatures). The presence of Al_4C_3 phase did not record in this experimental material. This fact can be caused either by incomplete transformation Al and C into Al_4C_3 during the preparation of material, consequently the effective amount of Al_4C_3 particles is lower that 4 vol.%, or by accomplished homogeneous distribution of very fine Al_4C_3 particles in Al matrix. Indexed phases in Fig. 1a,b confirmed according to JCPDS reference file: aluminium 04-487, platinum 04-0802.

Since plastic deformation during extrusion process took place in the aluminium matrix, we chose the aluminium diffraction peaks to calculate the TC. The XRD spectra of the samples in both directions, Fig. 1a,b, exhibit reflections due to (111), (200), (220), (311) and (222) planes of the aluminium, the degree of orientation of different planes is not similar. The measured intensities of individual aluminium diffraction peaks in these figures are not conformable with the relative intensities of aluminium standard but they are markedly different, what suggests that after plastic forming – extrusion the crystals are not randomly arranged and material is characterized by preferred orientation, or texture. The calculated values of the TCs are in Table 1a,b. For a preferentially oriented sample,

Table 1. Texture coefficients of Al-4vol.%Al_4C_3 alloy measured at different temperatures in both extrusion directions

a) radial	Al-4vol.%Al_4C_3					
	Temperature (K)						
hkl	298	373	473	573	673	773	298 a. c.
	TC						
(111)	1.79	1.75	1.89	1.89	2.08	1.85	1.54
(200)	0.77	0.75	0.81	0.81	0.92	0.80	0.69
(220)	0.04	0.04	0.05	0.04	0.06	0.07	0.14
(311)	0.11	0.09	0.04	0.04	0.16	0.15	0.23
(222)	2.3	2.33	2.15	2.15	1.79	2.11	2.4

b) axial	Al-4vol.%Al_4C_3						
	Temperature (K)						
hkl	298	373	473	573	673	773	298 a. c.
	TC						
(111)	0.27	0.31	0.31	0.31	0.40	0.43	0.38
(200)	0.68	0.76	0.70	0.79	0.82	0.92	0.72
(220)	3.09	2.95	3.06	2.93	2.77	2.71	2.79
(311)	0.73	0.79	0.73	0.78	0.76	0.64	0.79
(222)	0.23	0.18	0.18	0.19	0.26	0.30	0.31

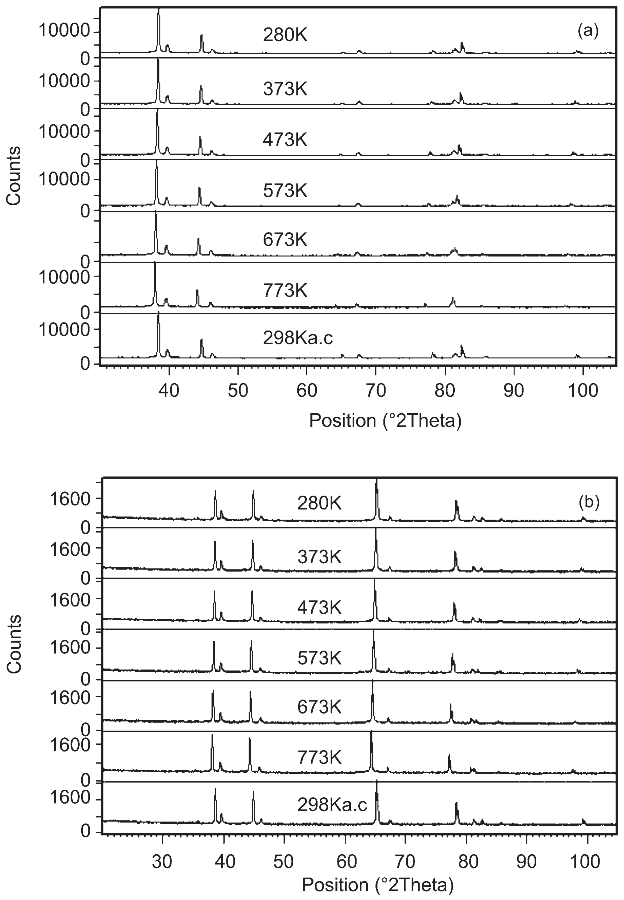

Fig. 2a,b. XRD spectrum of Al-4vol.%Al_4C_3 scanned at different temperatures: a) in radial extrusion direction, b) in axial direction.

the texture coefficient $TC_{(hkl)}$ should by greater than one.

In the radial direction of this material TC_{111} and TC_{222} are the largest and in the axial direction TC_{220} is the largest. The planes (220) and (222) are the secondary reflection planes of (110) and (111), respectively. In the radial direction is consequently Al (111) and in the axial direction is Al (110) the dominating crystal orientation.

The plastic deformation is carried out through the movement of dislocations. Aluminium has the cubic face-centred (fcc) lattice, which is characterized according to the slip mechanism theory by the basic slip plane (111) and slip direction ⟨110⟩. The slip system will rotate around the force axis during deformation. The movement of a crystal in poly-crystal structure is more complicated process than in the case of a single crystal because the crystal needs to accommodate the deformation of the neighbouring crystals during all the movement including rotation. Each slip plane has a general trend to rotate and to slope to the place perpendicular to compressive stress. In aluminium (111) planes rotate and slope to radial face and make TC_{111} higher than other texture coefficients [4].

High values of texture coefficients for (111) plane in the radial direction and for (110) plane in the axial direction indicate the presence of markedly deformed texture in material. The presence of 4 vol.% very fine Al_4C_3 particles in Al matrix is not able to prevent aluminium crystalline lattices rotation during mechanical work – extrusion. This amount of secondary phase so cannot prevent the texture formation under the used conditions for extrusion in our experimental alloys.

3.1.2. Measurements at elevated temperature

Figure 2a,b presents the comparison of the XRD spectrum in radial and axial direction for extruded Al-4vol.%Al_4C_3 material, obtained at different temperatures (ambient, elevated to 773 K and after cooling to ambient temperature). From Fig. 2a,b is evident, that the intensities proportions of individual aluminium reflections in extruded materials are approximately identical at complete measured temperature range, what implies that during and after thermal loading the texture is unchanged. The calculated values of the TCs at single temperatures are documented in Table 1a,b. The Al (111) orientation in the radial direction and Al (110) orientation in the axial direction remain unchanged within the range from 373 to 773 K and after cooling to ambient temperature in comparison to the sample after extrusion.

In the alloy with 4 vol.% Al_4C_3 strong extrusion texture was developed at used conditions of extrusion. The texture after extrusion is stable to 773 K.

3.2. Al-12vol.% Al_4C_3 material

3.2.1. Measurements at ambient temperature

Figure 3a documents the diffraction lines course in axial direction for extruded experimental Al-12vol.%Al_4C_3 material at ambient temperature and Fig. 3b presents the diffraction lines course in radial direction. The qualitative phase analysis of these spectra confirmed the lines of aluminium, platinum and aluminium carbide. Indexed phases in Figs. 1 and 3 confirmed according to JCPDS reference file: aluminium 04-487, platinum 04-0802 and aluminium carbide 01-0953. On the contrary, for the Al-4vol.%Al_4C_3 the intensities of aluminium diffraction lines in Fig. 3a,b approach the intensities of Al standard and the degree of orientation of different planes in both directions is similar. Calculated values of the texture coefficients for this material in both directions are shown in Table 2a,b. The TC_{111}

Table 2. Texture coefficients of Al-12-vol.%Al_4C_3 alloy measured at different temperatures in both extrusion directions

a) radial — Al-12vol.%Al_4C_3

	Temperature (K)						
hkl	298	373	473	573	673	773	298 a. c.
	TC						
(111)	1.39	1.35	1.39	1.32	1.30	1.32	1.35
(200)	0.89	0.81	0.83	0.84	0.83	0.84	0.89
(220)	1.13	1.09	1.19	1.07	1.18	1.13	1.09
(311)	0.81	0.61	0.81	0.83	0.75	0.80	0.68
(222)	0.79	1.16	0.79	0.93	0.92	0.90	0.96

b) axial — Al-12vol.%Al_4C_3

	Temperature (K)						
hkl	298	373	473	573	673	773	298 a. c.
	TC						
(111)	1.32	1.33	1.37	1.35	1.35	1.47	1.40
(200)	0.92	0.91	0.96	0.92	0.92	0.88	0.96
(220)	1.07	1.08	1.00	0.99	0.99	1.07	1.03
(311)	0.76	0.89	0.86	0.78	0.78	0.74	0.82
(222)	0.93	0.76	0.78	0.96	0.96	0.84	0.80

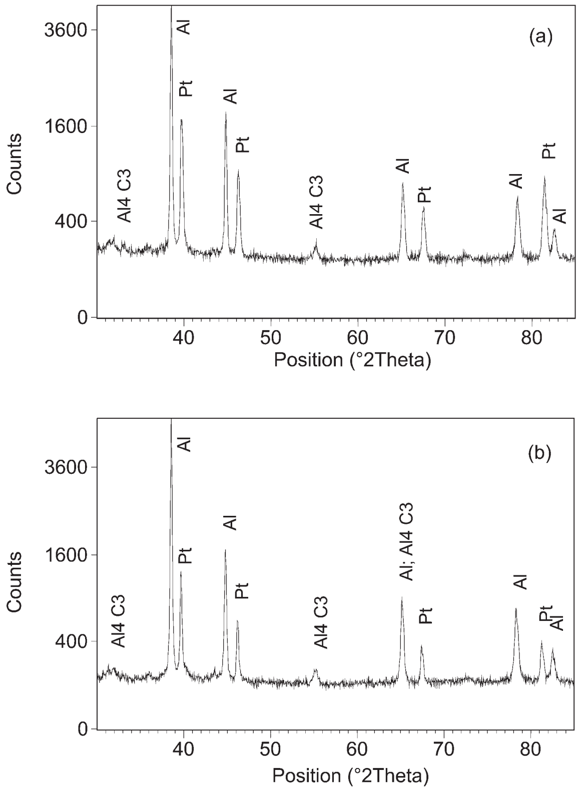

Fig. 3. XRD spectrum of Al-12vol.%Al_4C_3 scanned at ambient temperature and the phase analysis: a) in radial extrusion direction, b) in axial direction.

values in both directions are highest, but all texture coefficients in this material approximate 1. It is possible to say that the used conditions for extrusion process have induced a very low intensity aluminium deformation texture in the experimental alloy. The presence of larger amount of Al_4C_3 particles would prevent crystalline lattices rotation during mechanical work, such as extrusion, and consequently the texture development. Therefore the larger amount of Al_4C_3 particles in Al matrix produces a barrier that it hinders the deformation texture formation.

3.2.2. Measurements at elevated temperature

The X-ray diffraction lines of sample Al-12vol.%-Al_4C_3 in radial and axial extrusion direction measured at several temperatures are shown on Fig. 4a,b. The intensities proportions of individual aluminium reflections in extruded materials are approximately identical in complete measured temperature range. Calculated values of the texture coefficients for this material in both directions are shown in Table 2a,b. The presence of 12 vol.% Al_4C_3 particles in Al matrix may hinder and even block grain boundary movement and suppress the deformation texture formation during extrusion as well as the recrystallization texture formation during temperature loading to 773 K of extruded material [17].

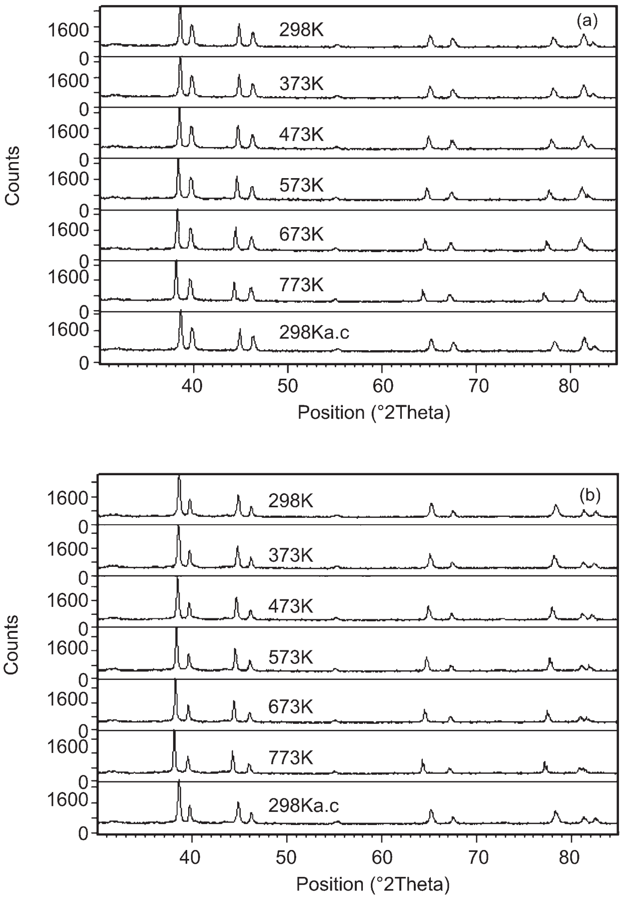

Fig. 4. XRD spectrum of Al-12vol.%Al_4C_3 scanned at different temperatures: a) in radial extrusion direction, b) in axial direction.

4. Conclusions

Dispersion strengthened aluminium alloy containing 4 vol.% Al_4C_3 shows after extrusion strong texturing for (111) planes in the radial direction and for (110) in the axial direction. The texture after deformation is stable up to 773 K.

Dispersion strengthened aluminium alloy containing 12 vol.% Al_4C_3 under the used conditions for plastic forming-extrusion process have induced a very low intensity aluminium deformation texture in the alloy. This volume of dispersoid particles efficiently hinders the deformation texture formation during mechanical work. The use of 12 vol.% Al_4C_3 dispersoid phase in aluminium alloy ensures suppression of structure recrystallization and obviously the recrystallization texture formation during extrusion process as well as at temperature loading of extrudate up to 773 K.

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