Plastic deformation of $Al_{85}Ni_{10}La_5$ by Equal Channel Angular Pressing

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Abstract

Helium-atomized amorphous $Al_{85}Ni_{10}La_5$ (at.-%) powder was investigated in the as-atomized state and after consolidation at different temperatures using equal channel angular pressing. Structural investigation by X-ray diffraction of as-atomized powder points at the presence of clusters with size of 1-2 nm. Equal channel angular pressing leads to the formation of fcc-Al precipitates with sizes between 10 nm and 13 nm for processing temperatures below 240°C, indicating a nucleation-controlled process. The results suggest a combined influence of shear deformation and temperature on the precipitation of fcc-Al crystals.

Key words: Aluminum alloys, metallic glass, crystallization, atomization, equal channel angular pressing, plastic deformation

1. Introduction

Aluminum-rich metallic glasses have attracted much interest due to their potential to develop nanostructured materials, which can have two to three times larger tensile strength in comparison with conventional polycrystalline Al-alloys. An increase in fracture tensile strength from 735 to 1010 MPa has been observed in Al-Fe-Gd ribbons after partial devitrification [1]. This precipitation hardening effect occurs, whenever a high number density of Al-nanocrystals ranging from 10^{21} to 10^{23} m^{-3} is present in the amorphous matrix [1-2]. Such Al-nanocrystals have been detected after heat treatment [3] and after plastic deformation [1, 4-5] of Al-based amorphous alloys. Deformation-induced crystallization at ambient and sub-ambient temperatures has been observed after cold-rolling [4], nanoindentation [5], high pressure torsion [6], extreme bending [7-8], and ball-milling [4, 9]. It is yet unclear whether the underlying mechanism for strain-induced crystallization is thermal or athermal. Since Al-rich metallic glasses are marginal glass formers, they can only be synthesized by non-equilibrium processes such as melt spinning, gas atomization, ball milling, etc. Therefore, these glasses can be produced only in limited sample dimensions such as powders, flakes or ribbons, requiring consolidation to a bulk material for any structural application. Consolidation by hot pressing [10] and hot extrusion [11] has to be performed at rather high temperatures (around the crystallization onset and higher) to achieve sufficiently compacted samples with low porosity. Due to the high processing temperature, the bulk materials consolidated by one of these processes are usually completely crystallized and become brittle due to the high content of intermetallic phases and exhibit poor mechanical properties. Spark sintering can be successfully performed at lower temperatures [12-14]. However, this yields specimens of high porosity. Alternatively to these methods, severe plastic deformation techniques such as equal channel angular pressing (ECAP) and high pressure torsion (HPT) can be used for powder consolidation at temperatures below T_x (T_x: Crystallization temperature in the as-atomized state) in order to avoid uncontrolled crystallization [6, 15-16]. While powder consolidation by HPT yields splats only with lateral dimension of centimetres and thickness in the order of 0.1 mm [15, 17], ECAP allows for powder consolidation of larger samples. In addition, ECAP can be performed continuously [18], which makes it an interesting candidate process for use in industry.

2. Experimental

$Al_{85}Ni_{10}La_5$ powders were gas-atomized by means of the NANOVAL process [19]. Master alloys were synthesized in an inductively heated furnace under Ar atmosphere using pure elements Al(99.98%), Ni(99.7%) and La(99.7%). The melt was heated up to approximately 1200°C and was subsequently helium atomized. Two batches of about 750 g powder per batch were produced from 1 kg $Al_{85}Ni_{10}La_5$ master alloy. A residue of alloy and slag remained non-atomized in the melting furnace. Granulometry of as-sprayed powder using a CILAS 715 particle size analyzer yielded uni-modal powder particle distributions with mean particle diameters of $\boldsymbol{d}$ = 11-12 µm for both batches. Particles with $\boldsymbol{d} < 2$µm could not be detected for technical reasons. Due to the similarity of the two particle size distributions for both batches, we will not distinguish between the two batches in the following. The composition of the as-atomized powders was measured by inductively coupled plasma mass spectroscopy (ICP-MS) assuming the presence of only Al, Ni and La in the sample material. The intensities of Al and Ni lines relative to La were determined and a mean value was calculated from 30 measurements per sample. The composition of the powders was found to agree with the nominal composition within 0.3 at. % with an estimated uncertainty of the measurements of ~0.1 at.-% [15].

Powder consolidation by means of ECAP was performed as illustrated in Fig. 1. Prior to ECAP, as-atomized powder was cold isostatically pressed applying an isostatic pressure of 1 GPa for about 2 min. The isostatically presssed samples were machined to fit into Cu containers and inserted into the pre-heated die and kept for 5 min before being pressed through the ECAP die. Details about the ECAP procedure of this work can be taken from Refs. [20-21]. According to Ref. [22], the ECAP die configuration used in this work yields an equivalent strain ε_{eq} of 1.15. Six ECAP samples were produced using different processing temperatures ranging from 175°C to 275°C. In the following, these samples are denoted as ECAP-X (X = 175, 200, 225, 240, 250, 275). Structure and microstructure were characterized by X-ray diffractometry (XRD) in a Bruker D8 Advance diffractometer using Cu K_α radiation (λ = 0.154 nm), and by scanning electron microscopy (SEM) using a Philips XL30 ESEM and a Zeiss Cross-Beam 1540ESB. Bragg peak positions of the diffraction data were analyzed by search and match method using PDF-2-data of the International Centre for Diffraction Data. The average crystal size was calculated from full-width-at-half-maximum (FWHM) of fitted peaks by use of Scherrer's formula. The volume fraction of fcc-Al phase in partially crystallized samples was calculated from XRD patterns by dividing peak areas of fcc-Al Bragg reflections with the scattering intensity of the amorphous phase. Continuous and isothermal heating experiments were carried out using a Perkin-Elmer differential scanning calorimeter (DSC) Pyris 1 under a constant argon purge. After the first run of each DSC measurement, a second run under the same conditions was performed on the fully crystallized sample in order to determine the baseline. The baseline was subtracted from first run data.

3. Results and Discussion

Load and ram speed vs. ram propagation during ECAP at 225°C are shown in Fig. 2. Range I reflects the compression of the Cu container only, while range II denotes the compression of the powder through the ECAP die. During the compression of the powder, the load varies between 500 MPa and 650 MPa while the ram speed varies between 1.2 mm/s and 1.7 mm/s.

Fig. 3 shows the cross-section of an ECAP-sample, which was stopped halfway in the ECAP die, along with SEM micrographs of different areas of the specimen. A big cavity is visible in the sample front between the powder and the Cu-container indicating that pores have been pressed towards the sample front where they coalesced during consolidation. In addition, many cracks have formed throughout the sample almost parallel to the container front. Apparently, ECAP did not yield consolidated powder throughout the whole sample. However, consolidation of powders was successfully achieved in crack-free regions of ECAP samples.

A qualitative inspection of the SEM micrographs reveals that the powder particles are slightly elongated in the upper part (point A) of the entrance channel. The particles are stretched and rotated along the ECAP channel. Taking the aspect ratio of the particles as a measure for plastic deformation, the evolution of deformation along the ECAP channel was analysed, see Fig. 4. The powder particles became more elongated from point A to point B. Starting from point C, the elongation first increases and then decreases when approaching point D. These changes in particle elongation reflect the deformation levels in the die. Obviously, the deformation level is highly non-uniform and is highest in the corner near point C. The high deformation in the die corner is ascribed to the change in direction of particle movement. The decrease in elongation near D might be due to the relatively large number of cracks and voids near point D which contributes to stress relaxation in the die.

Even at the highest deformation levels the powder particles can be distinguished from each other, indicating that the particles were not fused during deformation. Higher levels of plastic deformation at room temperature by high pressure torsion of the same powder yields fully consolidated samples with fused particles [15]. According to ref. [15], an equivalent strain of $\varepsilon_{eq} > 3$ is required to merge $Al_{85}Ni_{10}La_5$ powder particles. Thus, the value of $\varepsilon_{eq} = 1.15$ used in our ECAP die was not sufficient to merge the powder particles.

The strong non-uniform deformation characteristics of our specimens is reflected by large cracks in the exit channel and by changes in particle shape and orientation which might be changed to more uniform deformation and mould filling by application of back pressure which avoids the formation of cracks [23-26]. In the present experiment back pressure was not applied. In principle, harder container-materials would increase hydrostatic pressure and shear strain transmission to powders in the plastic deformation zone. However, ECAP experiments on amorphous and partially crystalline $Al_{87}Ni_8La_5$ and $Al_{85}Ni_{10}La_5$ powders using stainless steel and annealed low-carbon steel as container materials were not feasible [20] due to the limited available maximum load of the set up. Furthermore, ECAP of amorphous $Al_{85}Ni_{10}La_5$ powders below the glass transition temperature using a tool with parallel channels [21] did not yield macroscopic compaction because crack formation was even more severe in that case [20].

In order to investigate the influence of ECAP processing parameters on microstructure and thermal properties, XRD and DSC measurements were performed in crack-free regions of ECAP samples. Fig. 5 represents XRD patterns on ECAP samples processed at different temperatures. Except ECAP-275, the XRD patterns of all the samples show diffuse scattering near $2\,\Theta \approx 38°$ superimposed with fcc-Al Bragg reflections, indicating the presence of an fcc-Al phase within the residual amorphous matrix. Three Cu Bragg reflections are visible, caused by the Cu container used for the ECAP experiments. The intensities of fcc-Al Bragg reflections increase with increasing processing temperature, reflecting an increase of the volume fraction of fcc-Al phase with increasing processing temperature (Table 1). XRD patterns of ECAP-275 show Bragg reflections of fcc-Al, Al_3Ni and $Al_{11}La_3$ phases. Diffuse scattering near $2\Theta \approx 38°$ is no longer present, indicating fully crystallized material.

The XRD patterns shown in Fig. 6 were taken from the entrance and exit channels of the ECAP-225 and ECAP-240 samples. They show diffuse scattering near $2\Theta \approx 38°$ superimposed with fcc-Al Bragg reflections. The intensities of fcc-Al Bragg reflections in the exit channel are larger compared to the entrance channel, indicating a larger volume fraction of fcc-Al phase in the exit channel. For ECAP-225, the volume fraction of fcc-Al increases from 1.2 % to 2.6 %, and for ECAP-240 from 1.7 % to 4.0 %. Hence, both the deformation temperature and the deformation level significantly promote the precipitation of fcc-Al nanocrystals. This finding is supported by results of high pressure torsion experiments on the same material [15]. Isothermal and continuous annealing of as-atomized powders used in our

ECAP experiments did not result in primary crystallization of fcc-Al [15, 20]. Instead, fcc-Al and a metastable phase are formed in a first stage and a mixture of different phases, including the intermetallic phases Al_3Ni, $Al_{11}La_3$ and fcc-Al in a second. These results support the idea that the underlying mechanism for primary crystallization of Al during ECAP is induced by both temperature and plastic deformation. In literature, the formation of fcc-Al nanocrystals during plastic deformation was often described by athermal mechanisms [6-7, 27-30]. The rise in macroscopic temperature during ECAP of different Al alloy powders at strain rates smaller 18 mm/s was considered to be far below 100 K [23]. The strain rates used in our experiments are one order of magnitude smaller (ram speed 1.2 mm/s – 1.7 mm/s). On the other hand, the tensile strength of amorphous Al-alloys is appreciably higher than those of pure Al and Al alloys resulting in higher temperature rises than for pure Al and Al alloys. From our XRD results, where intermetallic phases were only detected after ECAP-275 we conclude a mean temperature rise during ECAP of at most 80 K. Fcc-Al precipitates in amorphous $Al_{85}Ni_{10}La_5$ powder are also produced during plastic deformation at room temperature as observed after HPT [15]. Crystallization caused by a local temperature rise in the shear bands during plastic deformation would yield a mixture of phases including fcc-Al, a metastable phase and, at higher temperatures, also the intermetallic phases Al_3Ni and $Al_{11}La_3$ rather than the formation of fcc-Al phase only.

Our results show that plastic deformation of amorphous $Al_{85}Ni_{10}La_5$ powder by ECAP at temperatures below 240°C results in a crystallization sequence different from those obtained by thermal treatment. These results are in qualitative agreement with results obtained by plastic deformation of amorphous $Al_{85}Ni_{10}La_5$ powder by high pressure torsion [15] and ball-milling [9].

Under all of these deformation procedures, fcc-Al precipitates with diameters in the order of 10 nm are formed. None of the stable or metastable intermetallic phases formed during thermal treatment was observed after plastic deformation. These observations point at nucleation-controlled primary crystallization under plastic deformation [2].

Recently, Lee et al. studied the influence of uniaxial pressure and plastic deformation of a Cu-based metallic glass [31]. They found that hydrostatic pressure reduces the activation energy for nucleation, ΔG_c, while shear stress lowers the activation energy ε for diffusion of a matrix atom to the nucleus, thus leading to an increase in the driving force for crystallization [31]. In ECAP experiments, both hydrostatic and shear stresses are applied. During plastic deformation the stresses are concentrated in the shear bands. We therefore assume that ΔG_c and ε decrease during ECAP processing of $Al_{85}Ni_{10}La_5$ powder, while the driving force for crystallization increases, resulting in deformation induced precipitation of fcc-Al in amorphous Al-based matrix. During ECAP, the system is steadily driven away from local equilibrium by dynamic input of strain energy in shear deformation zones (SDZ) [2], thus resulting in a decrease of ΔG_c and an increase in driving force. ε decreases due to increasing free volume localized in shear bands [32-34]. Additionally, adiabatic increase in temperature in the SDZs promotes nucleation. It has to be noted that classical nucleation theory disregards the fact that quenched-in nuclei and/or other structural inhomogeneities are already present in the powder. Such inhomogeneities may act as sites for stress localization, which promote crystallization within the amorphous matrix.

Deformation-induced crystallization of fcc-Al results in compositional change of the residual amorphous matrix, reflected by the changes in the DSC measurements above 300°C, see Fig. 7a. The exothermic heat flow below the glass transition temperature T_g, assigned to structural relaxation of the amorphous phase, gradually disappears with increasing ECAP processing temperature, see Fig. 7b. This is probably due to structural relaxation within the amorphous phase at higher processing temperatures. ECAP samples processed in the temperature range between 175°C and 225°C still show a glass transition which is, however, less pronounced compared to the as-atomized powder. Endothermic heat flow peaks are similar in ECAP-175 and ECAP-200, and slightly decreased in ECAP-225. The glass transition disappears when larger ECAP processing temperatures are applied. At room temperature, plastic deformation by ball-milling or high pressure torsion of the same alloy powder was shown to result in leading to enhanced heat release below T_g compared to the as-atomized powder accompanied by relaxation of excess free volume induced by plastic deformation, thus masking the endothermal reaction of the glass transition [35-36]. The decrease in exothermal heat release below T_g with increasing ECAP temperature points to the annihilation of free volume during ECAP at these temperatures. One would therefore expect a more pronounced glass transition compared to as-atomized powder. The masking of the glass transition in ECAP must therefore be due to other reasons. It is probably due to a composition dependence of the glass transition. An increase in the amount of deformation along with an increase in processing temperature enhances the formation of fcc-Al nano-precipitates thus resulting in an Al-depletion of the residual amorphous matrix. Obviously, the alloy loses its glassy nature with increasing Al-depletion. The gradual disappearance of the glass transition during plastic deformation is therefore ascribed to gradual changes in the composition of the residual amorphous matrix with increasing deformation level and temperature. Fcc-Al nanocrystals are also formed during plastic deformation at room temperature. The related change in composition is, however, small compared to deformation at elevated temperatures. Therefore, the relaxation of free volume induced by plastic deformation at room temperature dominates the heat release.

Furthermore, DSC curves of ECAP samples processed at different temperatures show that the exothermic heat release between 260°C and 290°C diminishes with increasing processing temperature (Fig. 7a). Their minima shift towards slightly larger temperatures. At the same time, the group of small exothermic events in the temperature range from 290°C to 350°C increase in peak area. A similar change in the DSC curve can be observed when going from the entrance ECAP channel to the exit channel of the ECAP die, see Fig. 8. These changes in the DSC curves are ascribed to the increase in volume fraction of fcc-Al with an increase in deformation level and with an increase in processing temperature. The exothermal reaction between 260°C and 290°C is mainly ascribed to the primary precipitation of fcc-Al nanocrystals. With increasing deformation level, the volume fraction of fcc-Al nanocrystals increases, thus leading to a decrease in exothermal reaction heat in the temperature range between 260°C and 290°C. The increasing volume fraction of fcc-Al nanocrystals is related to an enrichment of the residual amorphous matrix with La and Ni, thus favouring the crystallization of Al_3Ni and $Al_{11}La_3$. As shown in Ref. [15], the increasing peak area of exothermic events between 290° to 350°C is associated with a sudden growth of all three equilibrium phases fcc-Al, Al_3Ni and $Al_{11}La_3$. The increase in volume fraction of fcc-precipitates during ECAP processing with an increase in deformation level and temperature indicates that the primary, deformation-induced precipitation of fcc-Al nanocrystals is promoted by temperatures at least up to 240°C.

4. Summary and conclusions

Amorphous $Al_{85}Ni_{10}La_5$ powders have been produced by helium atomization. As-atomized amorphous powders reveal a glass transition which gradually vanishes with increasing deformation level. XRD on as-atomized powders points to the presence of quenched-in Al nuclei of 1-2 nm size. Plastic deformation of as-atomized amorphous powder yields crystallization products which are different from those obtained by thermal treatment. Fcc-Al nanocrystals with sizes of 10 nm - 13 nm embedded in a residual amorphous phase were detected after ECAP at temperatures far below the crystallization onset of as-atomized powder. The volume fraction of fcc-precipitates increases with the level of shear deformation and with the ECAP processing temperature, while the size of the deformation-induced fcc crystals hardly changes. The increase in volume fraction of the fcc-Al phase with the deformation level and temperature along with the absence of intermetallic phases after ECAP at sufficiently low temperatures suggests that deformation-induced crystallization of fcc-Al is not a purely thermal or athermal process but a combination of both. A quantitative characterization of the contributions from temperature and deformation to the formation of fcc-Al crystals requires a systematic variation of temperature and pressure which was beyond the scope of this work.

As the amorphous phase is not fully preserved during ECAP, plastic shear deformation, e.g. by ECAP, might be used as an alternative strategy to tailor nanostructured Al based materials.

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Figure Captions

Fig. 1
Schematic representation of the experimental procedure and setup of the ECAP process.

Fig. 2
Load (black curve) and ram speed vs. ram propagation during ECAP at 225 °C. Range I reflects pressing of Cu container only, while range II is related to pressing of powders through the corner of the ECAP die.

Fig. 3
Cross-section of an ECAP specimen stopped half-way using a processing temperature of 250°C. SEM images on the right hand side and at the bottom show enlarged sample areas. The red arrows denote the directions of the rotated powder particles.

Fig. 4
Aspect ratio and rotation angle α of particles along the paths A-B (a) and C-D (b) in Fig. 3

Fig. 5
XRD patterns of ECAP samples obtained at different temperatures. The Bragg reflections of Cu are caused by residual parts of the ECAP Cu container.

Fig. 6
XRD pattern of two specimens stopped halfway in the ECAP die where they were processed at 225 °C and 240°C, respectively. Two XRD patterns per specimen were recorded in specimen regions of the entrance and exit channel of the ECAP die.

Fig. 7
DSC curves of ECAP specimens obtained during continuous heating with 20 K/min (a) over large temperature range, and (b) over limited temperature range. Specimens processed at different temperatures. The steps in Heat Flow of ECAP-175 at 114°C and at183°C are due to irregularities in the electronics.

Fig. 8
DSC curves recorded from material taken from both the entrance and the exit channel of ECAP specimens processed at 225 °C and 240°. The ECAP process was stopped half way.

Table 1
Crystal size and volume fraction of fcc-Al phase determined from Fig. 5 by the use of Scherrer’s equation.