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Effect of Grain Size on Cryogenic Mechanical Properties of an Al-Mg-Sc Alloy

Daria Zhemchuzhnikova[a], Rustam Kaibyshev[b]

Belgorod State University, Pobeda 85, Belgorod 308015, Russia

[a]zhemchuzhnikova@bsu.edu.ru, [b]rustam_kaibyshev@bsu.edu.ru

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Abstract. An aluminum alloy with a chemical composition of Al–6%Mg–0.35%Mn–0.2%Sc–0.08%Zr–0.07%Cr (in wt.) and an initial grain size of ~22 μm was subjected to equal-channel angular pressing (ECAP) at 593 K up to a total strain of ~12. Extensive grain refinement provided the formation of fully recrystallized structure with an average grain size of ~0.6 μm. The mechanical properties of the alloy in two different structural conditions were examined at temperatures ranging from 77 to 293 K. It was shown that ECAP highly enhanced the strength, ductility and fracture toughness of the material over the wide temperature interval. Positive effect of grain refinement tends to increase with decreasing temperature due to suppression of brittle intergranular fracture. At ambient temperature, the extensive grain refinement provides +65% increase in yield stress (YS) and ductility, concurrently. At 77 K, YS increase is + 77%, and the ductility increase is +113% owing to grain refinement. Effect of the grain size on fracture toughness at cryogenic temperatures is discussed.

Introduction

Unique combination of strength and ductility makes aluminum alloys of the 5xxx series attractive for subzero temperature applications [1-3]. However, these alloys exhibit moderate strength and, therefore, the development of a high strength alloy belonging to these series are of crucial importance for realizing low weight cryogenic structures. Recent works [4,5] have shown that there is a great potential for a significant increase in strength of non-age hardenable aluminium-magnesium alloys through the formation of ultra-fine grained (UFG) structure using equal-channel angular pressing (ECAP). At present, there exists very limited information on the effect of extensive grain refinement on the mechanical properties of Al-Mg-Sc alloys at subzero temperatures, therefore, it is necessary to evaluate systemically the influence of UFG on this. The aim of the present work was to evaluate the effect of UFG on mechanical properties at cryogenic temperatures.

Experimental Procedure

The alloy, denoted here as 1575C Al, with a chemical composition of Al–6%Mg–0.35%Mn–0.2%Sc–0.08%Zr–0.07%Cr (in wt.) was manufactured by semi-continuous casting. The ingot was then homogenized at 633 K for 12 h and cut into plates with a rectangular shape and dimensions of 180 mm × 180 mm × 40 mm. These plates were processed by ECAP at 593 K up to a total of ~ 12, using route B_{CZ} [6] and a die with rectangular cross-section of 180 mm × 40 mm and channel inner angle of 90°. The details of sample preparation for structural characterization methods by optical metallography (OM), transmission electron microscopy (TEM), electron backscattering diffraction (EBSD) analysis were described in previous papers [7-9].

Tensile tests were carried out at 77–293 K on Instron 5882 testing machine equipped with an Instron 3119-408 cryogenic chamber, using specimens with a gauge section of 3 mm × 7 mm machined along the last pressing direction. Each sample was held at the testing temperature for about 10 min in order to reach thermal equilibrium. Charpy impact tests were carried out in the same temperature range on standard 10 mm × 10 mm × 55 mm specimens with a 2 mm U-notch in accordance with ASTM standard E23-05 using an Instron IMP460 machine with a capacity of 300 J. The longitudinal sections of the specimens were parallel to the last extrusion axis of the pressed billets. The impact values were averaged over three tests at each experimental point.

Results and Discussion

Microstructures before and after ECAP. The initial microstructure of the 1575C Al before ECAP consists of coarse grains having round shape with an average size of about 22 μm (Fig. 1a). The fraction of high-angle boundaries (HABs) and the average misorientation are 87% and 38°, respectively (Fig. 1a). The density of lattice dislocations is relatively low ($\rho \sim 3\times10^{12}$ m^{-2}) (Fig. 1b). Size of the coherent $Al_3(Sc,Zr)$ dispersoids ranges from 10 to 15 nm; these precipitations were uniformly distributed within the matrix, while the incoherent $Al_3(Sc,Zr)$ particles with an average size of 40 nm precipitated on boundaries [9]. The incoherent Al_6Mn dispersoids having plate-like or round shapes with an average thickness of 25 nm were observed within the grain interiors (Fig. 1b) [9].

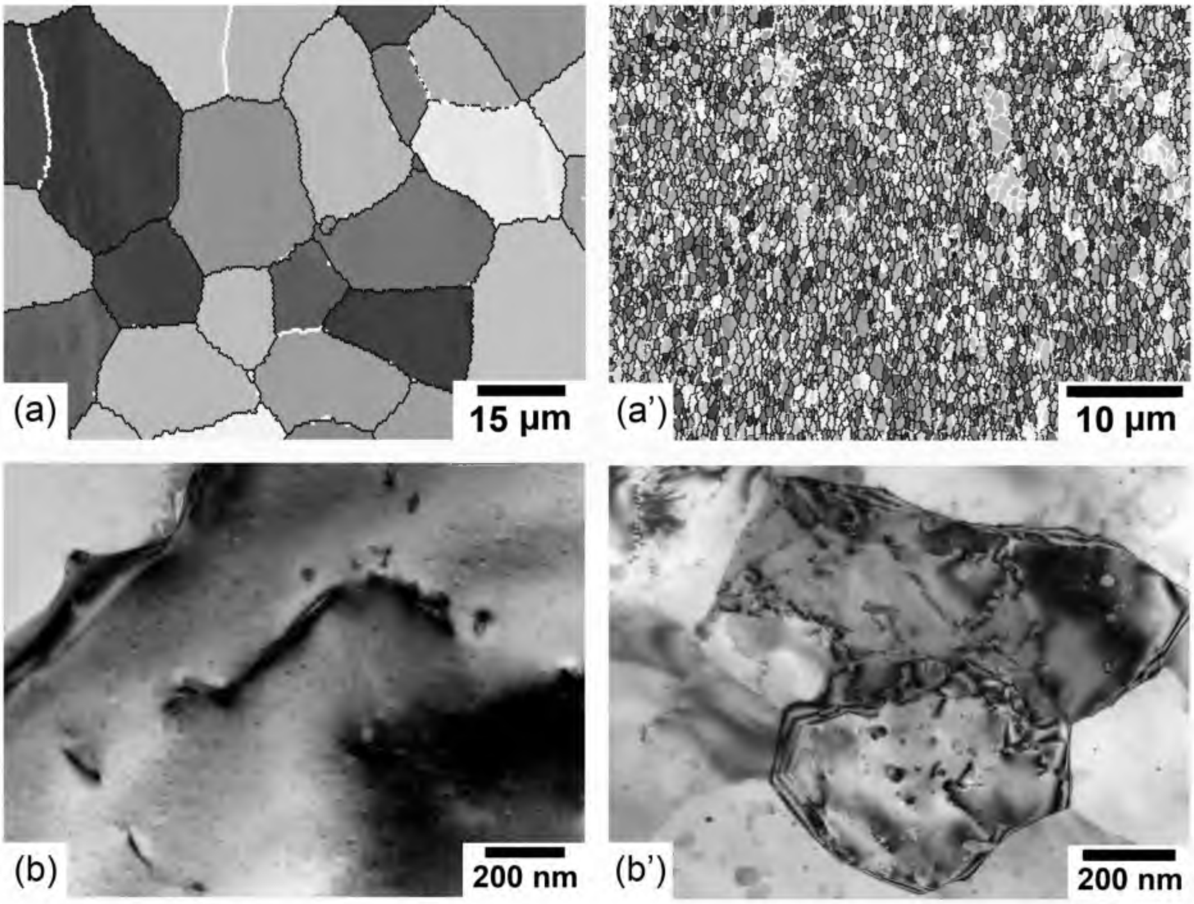

Fig. 1. Microstructures of the 1575C Al before (a, b) and after ECAP (a', b').

The ECAP led to the formation of almost uniform structure with an average grain size of ~ 0.6 μm and the recrystallized volume fraction more than 90% (Fig.1a'). The population of HABs and the average misorientation are 84% and 35°, respectively. The TEM study revealed a dispersion of coherent Al_3Sc dispersoids with an average size of ~12 nm and incoherent Al_6Mn precipitates with an average size of 32 nm and equiaxed shape. Both these particles are uniformly distributed within grain interiors. No remarkable effect of ECAP on distribution of second phase particles was found.

Most of grains contain moderate density of lattice dislocations ($\rho \sim 4\times10^{13}$ m^{-2}) which are pinned by precipitates (Fig. 1b'). Thus, ECAP up to $\varepsilon \sim 12$ provides extensive grain refinement by a factor of about 35; density of lattice dislocations increases slightly.

Mechanical properties. Fig. 2 shows the effect of temperature on the stress-strain curves and the Charpy U-notch impact energy of the 1575C Al in the both states. Magnitudes of yield stress (YS, $\sigma_{0.2}$), ultimate tensile strength (UTS, σ_B), total elongation (δ) and impact toughness (KCU) are summarized in Table 1. Extensive grain refinement affects mechanical behavior significantly. In the as-cast 1575C Al an extensive strain hardening takes place up to failure, while in the alloy subjected to ECAP the at subzero temperatures, the σ-ε curves exhibit well-defined upper yield point and the yield point elongation (Fig. 2a,b). In the as-cast alloy the flow stress tends to increase with decreasing temperature, while the ductility tends to increase until attaining a maximum at 153K and then decreases (Fig. 2a, Table 1). The serrated flow is manifested as the repeating oscillations on the stress–strain curves at 293 K and 223 K (Fig. 2a). This phenomenon is generally associated with the Portevin- Le Chatelier (PLC) effect attributed to dynamic strain aging [10,11].

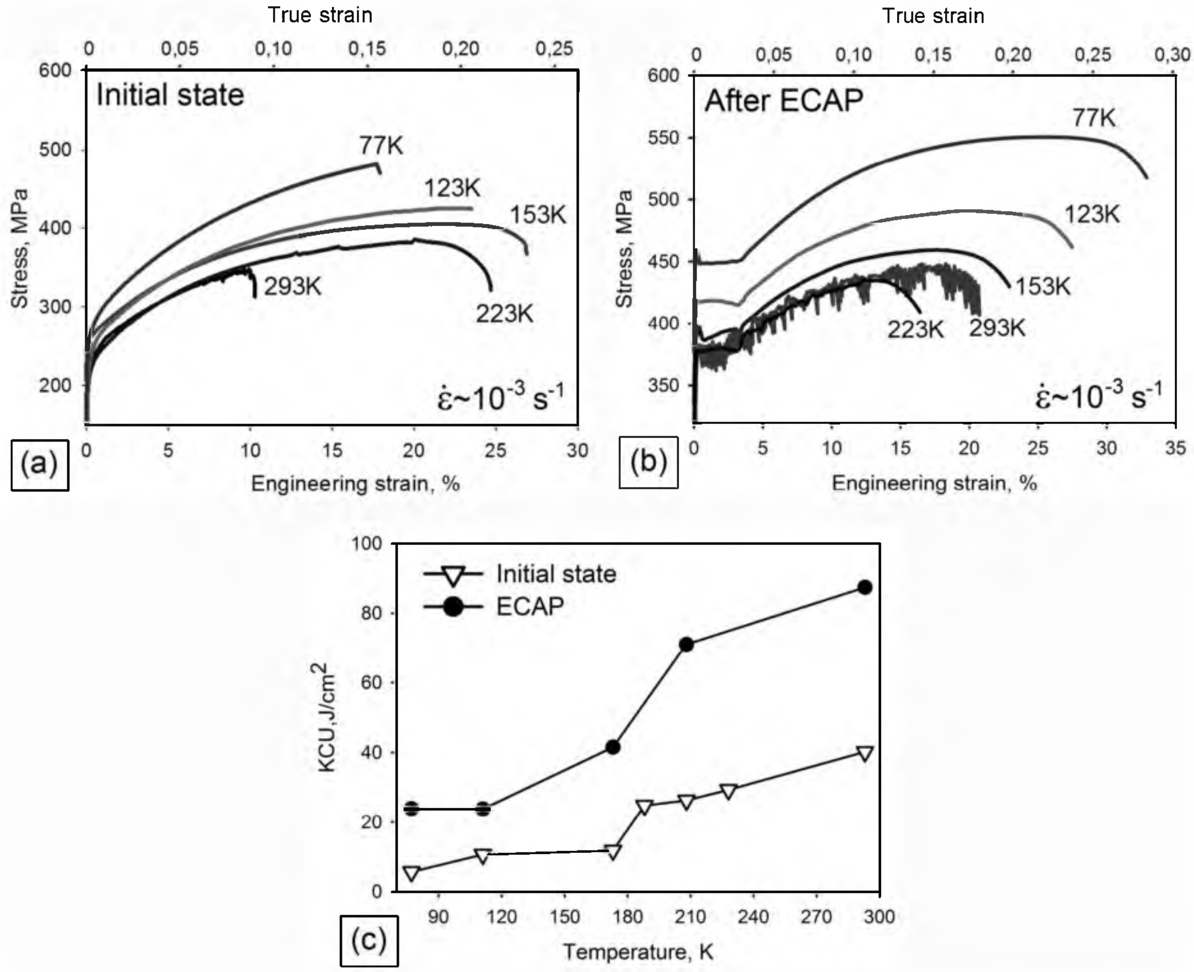

Fig. 2. Effect of temperature on the stress–strain curves (a, b) and on the impact energy (c) for the 1575C Al in initial state and after ECAP.

At ambient temperature, extensive grain refinement provides +65% increases in YS and ductility and +25% increases in UTS (Fig. 2b, Table 1). The flow stress and ductility of alloy subjected to ECAP tend to increase with decreasing temperature: at 77 K, YS increase is +77% and the total elongation increase is +113% in comparison with the as-cast alloy (Table 1). Therefore, increment in strength and ductility due to ECAP is higher at liquid nitrogen temperature rather than at ambient temperature. At room temperature, the as-cast alloy exhibits type E serrations and the 1575C Al with UFG structure demonstrates type B serrations (Fig.2a,b).

Table 1. Mechanical properties of aluminum alloy 1575C before and after ECAP.

Temperature (K)	Mechanical properties							
	Initial state				After ECAP			
	$\sigma_{0.2}$ (MPa)	σ_B (MPa)	δ (%)	KCU (J/cm^2)	$\sigma_{0.2}$ (MPa)	σ_B (MPa)	δ (%)	KCU (J/cm^2)
293	225	360	12	40.2	365	440	22	87.4
223	208	383	24	28.6	375	435	19	73.6
153	220	400	25	11.5	395	460	25	37.1
123	240	430	23	11.2	415	490	29	28.6
77	260	470	16	5.7	450	550	34	24

The effect of temperature on the Charpy U-notch impact energy is shown in Fig. 2c. The both states of the alloy exhibit a well-defined ductile-brittle transition (DBT) in the temperature interval 150–200 K (Fig. 2c). The impact toughness of the as-cast alloy drops from 25 to 12 J/cm2 and for T<180 K, the value of the Charpy U-notch impact gradually decreases to 6 J/cm2 with decreasing temperature (Table 1). The formation of UFG structure provides + 217% increase in the impact energy at room temperature. The impact toughness of alloy subjected to ECAP decreases with decreasing temperature (Fig. 2c, Table 1). However, even at 77 K, the value of the Charpy U-notch impact energy of the 1575C Al with UFG structure is 24 J/cm2, which is higher by a factor of ~ 4 in comparison with the as-cast alloy. Thus, ECAP extraordinary enhances fracture toughness of the 1575C Al. The positive effect of ECAP on the Charpy U-notch impact energy is superior at cryogenic temperatures.

Fractography. Fractographs of the fractured tensile specimens are shown in Fig. 3. At room temperature, the both materials shows dimple rupture associated with considerable plastic deformation [12] (Fig. 3a, a'). Fractography of the as-cast 1575C Al was described in previous work [9] in detail. Decreasing temperature leads to transition from ductile transgranular fracture to intergranular brittle fracture (Fig. 3b) [9]. In the material with UFG structure at room temperature, a number of dimples with deep conical shape are observed (Fig. 3a'). This fracture surface results from limited nucleation sites, which are widely spaced; the microvoids grow to a large size before coalescing which requires extensive plastic deformation (Fig. 3a'). The second phase particles are typically present at the bottom of most of large dimples. It seems that fracture is initiated by the separation of secondary phase particles from the aluminum matrix. Failure primarily occurs along a transgranular fracture path [11]. At 77 K, the fracture mode of the 1575C Al with UFG structure does not change significantly, while intergranular fracture of the unrecrystallized structure could be occasionally observed (Fig. 3b'). Ductile transgranular fracture is in dominant. In addition, at the cryogenic temperature, size of the microvoids, which were able to grow to large size, decreased dramatically. As a result, the formation of fracture surfaces requires more extensive plastic deformation comparing with room temperature. It is apparent, that boundary particles in the alloy with UFG structure do not serve as void-nucleating sites [12] at cryogenic temperature in contrast with room temperature. This results in increased ductility with decreasing temperature that is in contrast with the as-cast alloy.

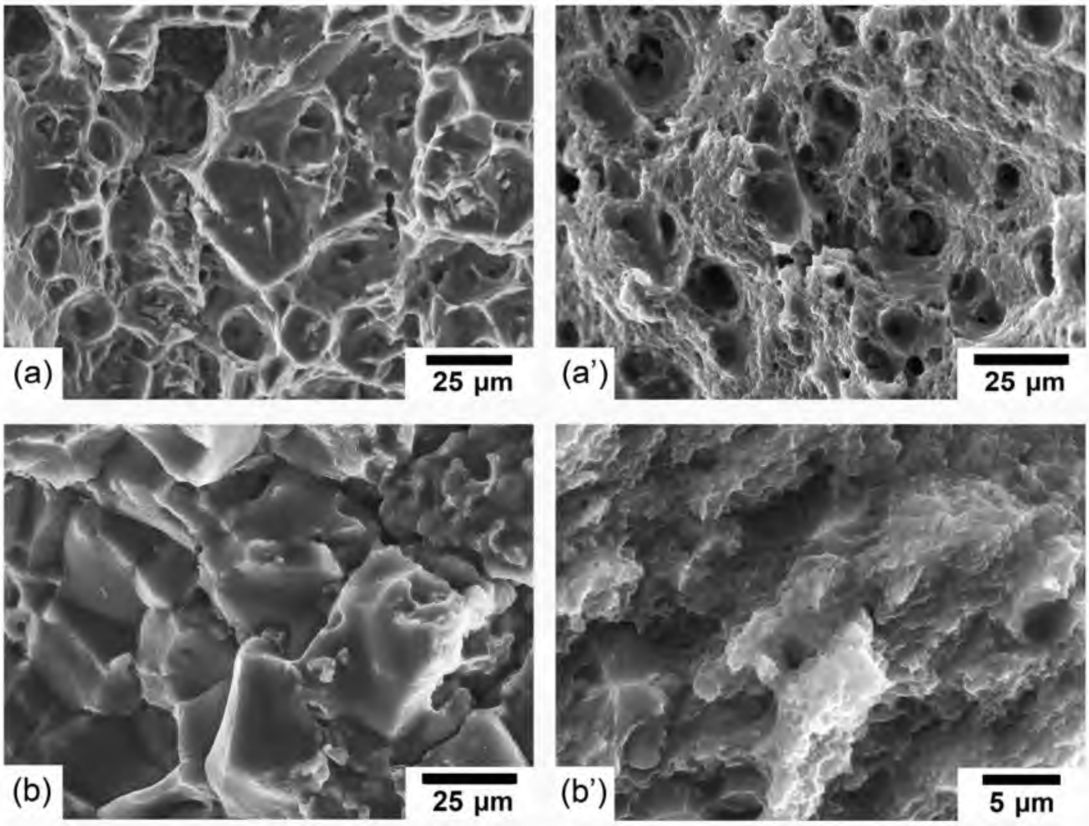

Fig. 3. SEM of fracture surfaces of the tensile specimens in the (a, b) as-cast state and (a’, b’) after ECAP deformed at temperatures: (a, a’) 293 K, (b, b’)77 K.

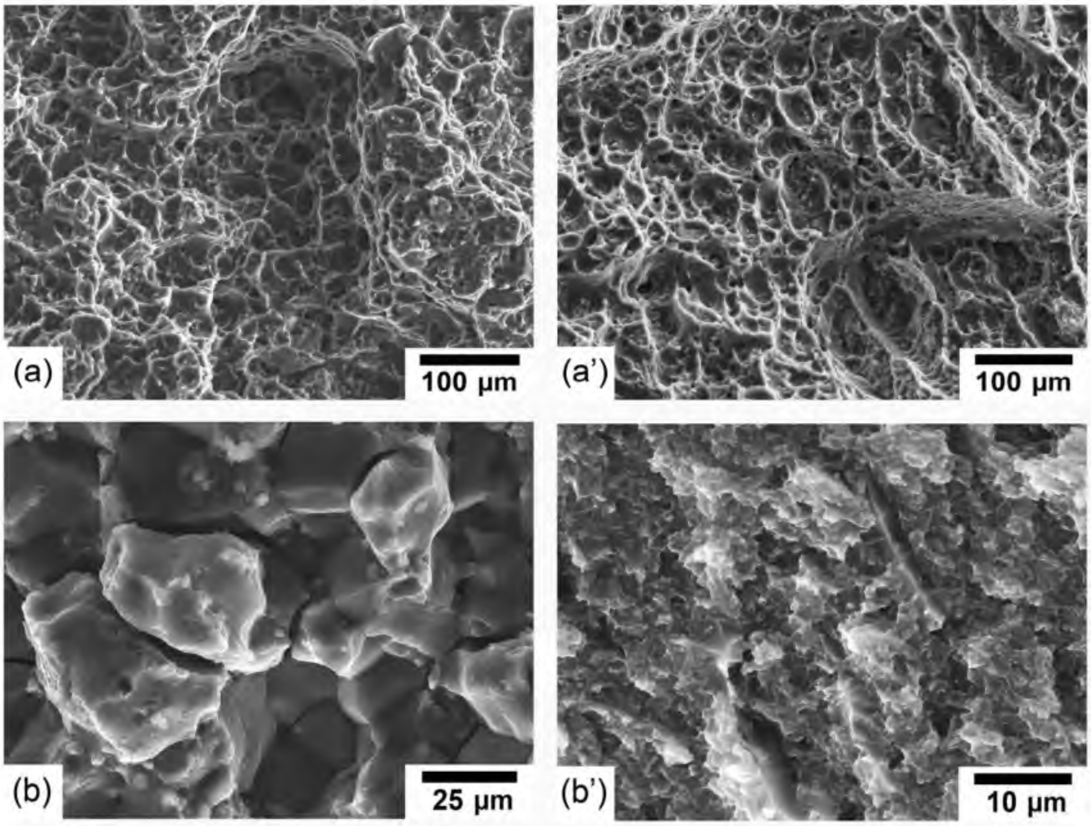

Fig. 4. SEM of specimens of 1575C Al in the (a, b) initial and (a’, b’) ECAP conditions after impact testing at temperatures: (a, a’) 293 K, (b, b’) 77K.

Inspection the fracture surfaces after the impact tests shows that, in general, the aforementioned DBT is attributed to the transition from ductile transgranular fracture (Fig. 4) to intergranular brittle fracture [9,12]. At 77 K, in the as-cast alloy the crack propagation along the boundaries induces cleavage of the grain interiors (Fig. 4b) [9,12]. Dimples indicating ductile transgranular fracture were rarely found (Fig.4a') [9]. Fracture at the liquid nitrogen temperature in the 1575C Al after ECAP has two main features. First, intergranular brittle fracture takes place occasionally along limited number of planar boundaries of unrecrystallized grains (Fig.4b'). Second, transgranular ductile fracture gives the main contribution to crack propagation (Fig.4b'). Numerous nucleating sites are activated and adjacent microvoids coalesce before they have an opportunity to grow to a larger size [11]. As a result, a large number of small dimples are observed and crack propagation requires considerable plastic deformation (Fig. 4b') [12]. As a result, the formation of UFG structure highly enhances fracture toughness.

Summary

Extensive grain refinement from ~22 to ~0.6 μm by ECAP at 573 K up to a total strain of ~12 highly improves strength, ductility and fracture toughness of the Al-Mg-Sc-Zr alloy at cryogenic temperature. At ambient temperature, the extensive grain refinement provides +65% increase in YS and ductility, concurrently, +25% increase in UTS, and + 217% increase in the Charpy U-notch impact energy. At 77 K, YS increase is + 77%, UTS increase is +17%, the ductility increase is +113% and the impact energy increase is +421%. The 1575C Al subjected to ECAP and in as-cast state exhibits a well-defined ductile-brittle transition in the temperature interval 150–200 K. However, even at 77 K, the value of the Charpy U-notch impact of the alloy with ultra fine grained structure remains high enough (24 J/cm^2).

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