Effect of deep cryogenic treatment on aging behavior and properties of Al-Mg-Si alloy

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The 6xxx alloys are one of the most commonly used aluminum alloys in automotive, electrical, aerospace, shipbuilding and off-shore plant industries due to very good corrosion resistance, anodizing properties and relatively good mechanical properties. Furthermore, these alloys are also easily extruded, welded and machined and are heat-treatable (homogenization and aging), allowing improvement of mechanical properties by aging after material forming. However, increased demands on strength, performance and carbon-footprint of materials used in modern design dictates development of new alloys as well as new environmentally friendly heat treatment strategies and processes. One of such processes, already successfully tested and proved on various steels, namely tool steels is deep cryogenic treatment (DCT). While mechanisms and effect of DCT for ferrous alloys are well investigated and described, DCT for non-ferrous alloys, such as the Al-Mg-Si aluminum alloys is still at its beginnings. Therefore, the aim of our research was to investigate the influence of DCT on aging behavior of 6xxx series aluminum alloys, how it effects microstructure and final alloy properties.

Keywords: *Deep Cryogenic Treatment, Al-Mg-Si alloy, aging, microstructure, hardness, fatigue*

1. Materials and methods

For this research commercially available EN AW 6026 alloy was used with the following chemical composition (in wt.%): 0.70% Mg, 0.68% Si, 0.66% Bi, 0.59% Mn, 0.34% Pb, 0.30% Cu, 0.27% Fe, 96.36% Al. Material in the form of test specimens was then subjected to different heat treatment procedures, which are schematically presented in Fig. 1. Firstly, the samples were separated into two major groups which were firstly homogenized for 1 h at different temperatures. The first group at 530 °C and the second at 570 °C. After homogenization the samples were quenched in water with an average temperature of 22 °C. After quenching half of the samples, denoted as deep cryogenic heat-treated (DCT), of both major groups were immersed in liquid nitrogen for 24 h or 48 h and afterwards warmed up to room temperature in ambient environment and finally aged. The other half of the samples, denoted as conventionally heat-treated (CHT), was directly aged after quenching. The aging for both CHT and DCT samples was performed at room temperature (natural aging) and at temperature of 190 °C (artificial aging). The natural aging was performed in a dry storage in ambient environment up to 28 days, whereas the artificial aging was performed for 8 h in a convection furnace. The time and temperature parameters of all heat treatment procedures is provided in Table 1 and Table 2, respectively.

Table 2 Heat treatment parameters for naturally aged samples

Samples		Homogenization		DCT		Natural aging	
		T (°C)	t (h)	T (°C)	t (h)	T (°C)	t (days)
Group A	w/o DCT	530	1	/	/	22	1, 6, 14, 21, 28
	DCT 24 h	530	1	-196	24		
	DCT 48 h	530	1	-196	48		
Group B	w/o DCT	570	1	/	/		
	DCT 24 h	570	1	-196	24		
	DCT 48 h	570	1	-196	48		

Table 3 Heat treatment parameters for artificially aged samples

Sample groups	Homogenization		DCT		Artificial aging	
	T (°C)	t (h)	T (°C)	t (h)	T (°C)	t (h)
1	530	1	/	/	190	8
2	530	1	-196	48		
3	570	1	/	/		
4	570	1	-196	48		

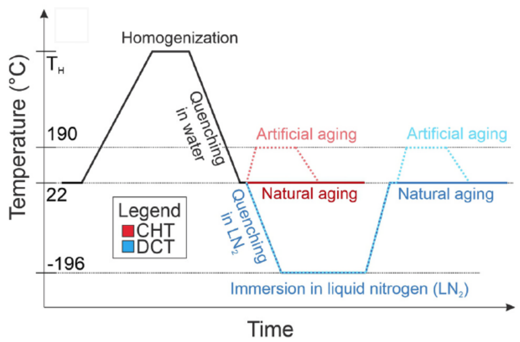

Figure 1 Scheme of the heat treatment procedure for AW 6026 samples

Hardness of the material after different heat treatments was measured according to the Brinell method (SIST EN ISO 6506–1:2014 standard; HBW 2.5/62.5), using Innovatest Nexus 7500 device. At least three measurements were made on each sample. For the naturally aged samples, the hardness was measured after 1 day, 6 days, 14 days, 21 days and 28 days after the homogenization (w/o DCT group) or DCT treatment (24 h DCT and 48 h DCT group). For the artificially aged samples, the hardness was measured after the complete heat treatment.

Tensile tests were performed using universal testing machine Instron 8802 with an Instron extensometer with initial gage length of 50 mm. Tensile tests were performed according to SIST EN ISO 6892–1:2017 standard using the A224 method and average results obtained from the measurements of three specimens

Impact toughness was measured with the Charpy impact test at room temperature according to international standard SIST EN ISO 148–1:2017 using CVN specimens and 300 J

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pendulum.

Fatigue behavior of the investigated EN AW 6026 alloy was tested under dynamic loading in bending mode using Rumul resonant fatigue testing machine Cracktronic with an operating frequency of around 180 Hz. The fatigue (S/N) curves were obtained by performing room temperature fatigue tests on standard Charpy V-notched (CVN) samples (10×10×55 mm) and using constant amplitude bending stress between 70 MPa and 120 MPa, stress ratio R of 0.1 and a sinusoidal waveform. Sample failure criterion was set as a drop of inherent oscillation by more than 3%, where the fatigue cracks occurred down to a depth of 3 mm.

2. Results and discussion

2.1 Natural aging

The hardness evolution during natural aging reveals an enhanced hardness increase with aging time. For the sample groups with lower homogenization temperature (Fig. 2(a)), a significant difference in hardness of 6 HB between the samples with and without DCT is already visible after 6 days of aging, despite the similar hardness values after 1 day of aging (around 76.5 HB). The initial difference between the samples is sustained throughout the remaining aging up to 28 days. Furthermore, the samples with 48 h exposure to liquid nitrogen (DCT 48 h) display a slightly faster hardness increase compared to the 24 h counterparts (DCT 24 h), which also yields a higher final hardness. In contrast to the first group, the group with higher homogenization temperature (Fig. 2(b)) displays a considerably lower impact of DCT on hardness development. The samples with and without DCT display a very similar hardness up to 14 days. Afterwards the DCT samples display a slightly higher hardness (about 2–3 HB) up to 28 days. By comparison of both sample groups, it is clear that the homogenization temperature has a considerable influence on the DCT effect on aging process of aluminum alloy EN AW 6026. Additionally, the less homogenized state (lower homogenization temperature) allows the development of higher hardness compared to the more homogenized state, when DCT is applied. In contrast, this is not the case when DCT is not applied.

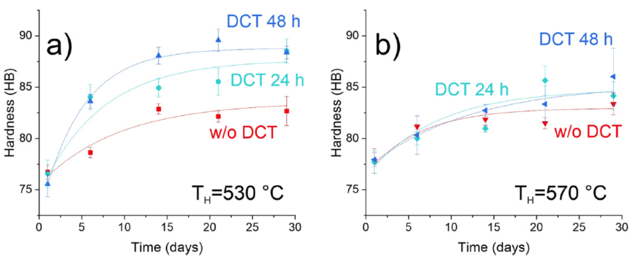

Figure 2 Hardness change over natural aging time for different heat treatments

2.2 Artificial aging

Results for artificial aging indicate that DCT has a positive influence on hardness (Fig. 3(a)) in the case of lower homogenization temperature (compare CHT 1 to DCT 2), whereas in the case of higher homogenization temperature the hardness levels are within the same range (compare CHT 3 to DCT 4). Contrary, the tensile properties are negligibly altered with DCT, when artificial aging is performed for samples with homogenization temperature of 530 °C (Fig. 3 (b)-(d)). Whereas for the second group the tensile strength (Fig. 3 (b)) and yield strength (Fig. 3 (c)) are raised with DCT and with it proportionally the elongation is reduced (Fig. 3(d)). Similarly as for the natural aging, these basic mechanical properties indicate that the homogenization temperature influences the DCT effect on the artificial aging of the two sample groups. The surprising feature of DCT effect for the first group is the increased hardness and simultaneous reduced tensile strength. Such behavior connected with the significantly higher hardness of the alloy in comparison to naturally aged samples indicates that the precipitation of β-type particles must be present. For the second group, the DCT samples display higher strength, but also a strong scattering of the properties from sample to sample in comparison to CHT samples (Fig. 4). The possible cause could be in the formation of Si particles that have a high notch effect, leading to higher chance of sudden failure with denser and finer precipitation for DCT samples. The impact toughness measurements, shown in Fig. 4 (a), indicate a slight increase in the mean value with DCT for lower homogenization temperature, whereas with higher homogenization temperature a slight deterioration of the property occurs with DCT. However, for both sample groups, DCT displays a negligible effect on the impact toughness, when scattering of the results is considered. Despite this, the obtained S-N curves from fatigue testing (Fig. 4 (b)) show a clear trend of improved fatigue resistance for DCT samples compared to their CHT counterparts. The significant improvement of the DCT sample as well as the modified shape of the S-N curve for the first group of samples correlates well with the simultaneous increase in hardness and impact toughness of the material. In the second group, the fatigue improvement at lower number of cycles is considered to be a result of the higher tensile strength after DCT that translates to higher resistance to fatigue at high loads.

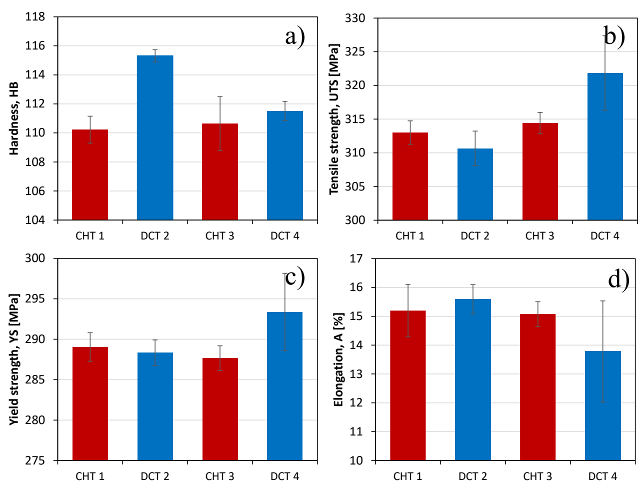

Figure 3 (a) Hardness, (b) tensile strength, (c) yield strength and (d) elongation of artificially aged samples

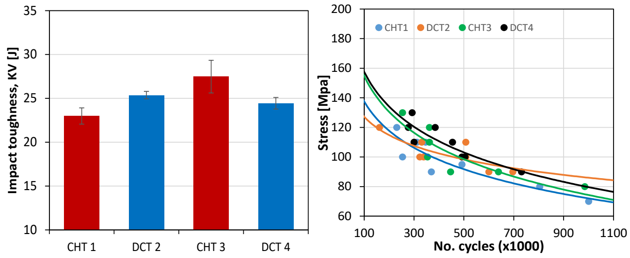

Figure 4 (a) Impact toughness and (b) fatigue properties of artificially aged samples

3. Conclusion

The effectiveness of DCT is strongly dependent on the homogenization temperature. The higher (570°C) homogenization temperature results in a significant reduction of the hardness improvement for DCT in comparison to the lower (530°C) homogenization temperature.

Due to the microstructural changes, the hardness change during natural and artificial aging is amplified with the application of DCT. The duration of exposure to DCT (from 24 to 48 h) also increases the impact of DCT on the hardness evolution during natural aging, increasing both the hardening rate and maximal level of achieved hardness.

Additionally, DCT yields improved fatigue resistance, especially with lower homogenization temperature, as well as increased strength, observed for samples with higher homogenization temperature.

The modification of mechanical properties after artificial aging with DCT can be partially related to denser precipitation of β'' particles and matrix depletion of Mg and Si.