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A research on the effect of retrogression and re-aging heat treatment on hot tensile properties of AA7075 aluminum alloys

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ABSTRACT

Aluminium alloys are preferred in most industries due to the functional properties they provide. It is known that alloys that can be processed with heat treatments shows better mechanical properties. 7xxx series alloys can be processed vi heat treatments and are often used in environmental conditions such as extreme temperatures and corrosive environments. Corrosive sensitivities such as stress corrosion cracking (SCC) can be observed with the effect of working conditions. It is known that retrogression and re-aging (RRA) heat treatment provide corrosion resistance and decrease the SCC velocity. The purpose of this study is to examine the tensile behaviour of annealed and retrogression-re-aging (RRA) heat treated AA7075 alloys at elevated temperatures. The mechanical properties of the alloys were investigated by conducting tensile tests at room temperature (RT), 100, 200, and 300°C. Hardness tests were performed at room temperature on the samples which were taken from tensile test specimens after tensile tests. The potential effects of test temperature on mechanical and microstructural properties were examined. The annealed and RRA heat treated alloys were characterized by scanning electron microscope (SEM), and X-ray diffraction (XRD) analysis. As a result, an increase in strength and hardness of the RRA treated AA7075

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alloys was observed. Ductility of the RRA alloy was lower compared to the annealed AA7075 alloy.

Fracture surface examinations showed that there was a semi-ductile fracture below 200°C and ductile fracture at temperatures of 200 and 300°C. Ductility was observed to increase with increasing temperature.

INTRODUCTION

Due to its favorable properties such as high strength, low density, and corrosion resistance, aluminum alloys are widely used in industrial fields such as aerospace and automotive. 7xxx series alloys are the most widely preferred aluminum alloys in the aerospace industry. Due to their mechanical and physical properties, as well as their clean, easy, and recyclable production, these alloys are well-suited for sustainable production processes. 7xxx series alloys contain between 1-8% Zn and 1-3% Mg in their chemical composition. Due to the high solubility of Zn and Mg, mechanical properties can be improved by heat treatments [1]. Aging heat treatment is commonly used for hardening aluminum alloys and improving their mechanical strength [2]. The hardness and strength of these alloys are increased by secondary phase precipitates formed in the microstructure and by obstructing/preventing the dislocation movement of these precipitates with the aging heat treatment [3,4]. The aging heat treatment is carried out in three stages. In the first stage, the alloy to be aged is taken into solution at a temperature below the eutectic point and dissolved. The second stage involves rapid cooling (quenching), creating a supersaturated solid solution. Finally, in the third stage, the process is completed by aging at a low temperature. The primary requirement of the aging heat treatment is that one or more alloying elements in the alloy is highly soluble in the matrix. The high dissolution level of Zn and Mg in 7xxx alloys allows them to be aged [5]. In aging stage, Zn and Mg cluster to form the second phase. Dislocation

movements that occur in the microstructure during deformation are forced by such secondary phases, and therefore an increase in strength occurs. Dissolved Zn and Mg atoms combine to form coherent and unstable Guinier–Preston zones (GP zones). During aging, more atoms cluster in GP zones, forming a semi-coherent η' phase. In later aging stages, the η' phase turns into a stable and incoherent η' MgZn₂ secondary phase [6]. The solution treatment temperature, aging temperature, and aging time are important parameters that affect the microstructure when aging AA7075 alloys. Tekeli et al. [4] investigated the effects of different solution temperatures (465-485°C) used to age the AA7075 alloy on the microstructure and mechanical properties of the alloy. The authors reported that an increase in the temperature of solution enabled the formation of secondary phase solutions, which led to an increase in hardness and strength. Şimşek et al. [3] studied the effects of T6 heat treatment applied for different aging times on the microstructure and mechanical properties of AA7075 alloys. The researchers aged the alloys between 15 hours and 35 hours, observed that the samples that were aged for 25 hours showed the highest hardness and tensile strength values, and reported that the hardness and tensile strength decreased when the alloys were aged further. Overaging techniques such as T73, T76 and T77 have been developed to improve certain properties of 7xxx alloys such as corrosion resistance. However, although these overaging techniques improve corrosion resistance, they also reduce strength significantly [7]. A technique known as retrogression and re-aging (RRA) has been developed to improve corrosion resistance while maintaining the strength properties of 7xxx series alloys [8]. Stress corrosion cracking (SCC) rate is known to be significantly

reduced by RRA heat treatment [9]. RRA heat treatment is the process of short-term re-aging of T6 treated 7075 alloy [10]. Fakioglu et al. [11] applied T6 and RRA heat treatments to annealed AA7075 alloys and observed that the increase in hardness and fatigue achieved by T6 heat treatment was maintained even if it was reduced by an acceptable amount by RRA heat treatment. Viana et al. [12] noted that the microstructural stability after re-aging was significantly affected by retrogression temperature. The authors reported that higher retrogression temperatures allowed for a more stable microstructure. Li et al. [13] observed that the heating rate had significant impacts on microstructure and mechanical properties in retrogression. The researchers reported that a moderate heating rate provided the most favorable mechanical properties. Ozer and Karaaslan [14] studied the effects of the RRA heat treatment applied at different re-aging temperatures and times on mechanical properties of the AA7075 alloy. According to the results of their study, increasing re-aging temperature and time lead to lower hardness.

Various studies were carried out to investigate the deformation mechanisms of 7xxx series alloys at elevated temperatures. It is known that as the aging temperature increases, the strength of alloys decreases, but some important mechanical properties such as toughness and ductility also improve. As a result of dynamic recovery in high temperature deformation processes in aluminum alloys, a sub-grain structure develops in the grains, which leads to strain hardening [15]. Grain boundary sliding leads to increased ductility by slowing down the formation of cracks in grains that have become ductile due to dynamic recovery [16]. Ductility is usually controlled by two important

material parameters. The first is the ability of the material to harden, and the second is the material's strain rate sensitivity [17]. Numerous studies were conducted on the effects of strain rate on high temperature deformation. Lei et al. [18] investigated the high temperature (350-500 °C) deformation behavior and microstructural properties of 7075 sheets for formability at high strain rates (in the range of 1×10^{-3} , $1 \times 10^{-2} \text{ s}^{-1}$). They observed that the peak stress decreased with increasing temperature and decreasing strain rate. In their tests conducted at all strain rates, they observed that the amount of percent elongation increased with increasing test temperature but showed a large decrease at high strain rates at 500 °C. Gupta et al. [19] performed compression tests on AA7010 and AA7075 alloys at elevated temperatures (between 350-450 °C) and different strain rates (0.001 to 10 s^{-1}). They observed that the peak stress decreased with increasing temperature and decreasing strain rate. Lei et al. [20] investigated the high temperature deformation properties of 7075 aluminum alloy sheets fabricated by twin roll casting and rolling. The researchers reported that second phase particles had significant effects on the ductility of alloys. It was noted that coarse particles (about $1 \mu\text{m}$) provided a fine-grained microstructure and high ductility and formability at high temperatures. Fine particles (about $0.1 \mu\text{m}$), on the other hand, caused a non-homogeneous microstructure, reducing the percent elongation at high temperatures. The researchers also reported that large particles ($>5 \mu\text{m}$) caused cracks in the particle and the particle-matrix interface, resulting in fractures at low percent elongation. Taheri-Mandarjani et al. [17] investigated the ductility behavior of extruded 7075 alloys at elevated temperatures (100-450 °C) and different strain rates (10^{-1} - 10^{-3} s^{-1}). They

reported that the peak stress decreased with increasing temperature at a certain strain rate. The researchers observed that high stress at low temperatures also depended on the presence of more precipitates in the structure at these temperatures, and that the effect of the strain rate on the peak stress at temperatures below 250 °C was negligible.

In the literature, most of the studies were carried out to investigate the high temperature formability of materials (mostly at high temperatures >400 °C) to determine the appropriate process parameters. Additionally, the studies generally focus on popular tempered alloys such as T6 or T651. Therefore, studies on the interaction of RRA heat treatment and mechanical properties at elevated temperatures are limited. The purpose of this study is to examine the mechanical properties of RRA treated AA7075 alloy at elevated temperatures. Annealed and RRA heat treated AA7075 alloys were subjected to tensile testing at room temperature, 100, 200, and 300 °C. In addition, hardness measurements were performed at room temperatures to investigate the effect of test temperature on hardness. Changes in the microstructure of materials deformed at elevated temperature were studied by scanning electron microscope (SEM). Secondary phases and compositions that can occur in the microstructure were studied by XRD analysis.

MATERIALS AND METHOD

Extruded AA7075 alloy rods were supplied from Alkor Casting Alloys Company. Table 1 shows the chemical composition of the AA7075 alloy used in experimental studies. All samples were first homogenized for 2 hours at a temperature of 500 °C. Protherm PLF110 heat treatment furnace was used for heat treatments of the samples. They were then prepared on the turning lathe in accordance with ASTM-E8M-04 and ASTM E21-09 standards. In the first stage, the prepared tensile samples were treated by T6 heat treatment. In the T6 heat treatment, all samples were quenched in water after 2 hours of applying solution treatment at 485 °C. Then the samples were aged for 24 hours at 120 °C. For the RRA heat treatment, the AA7075-T6 samples were solution treated again at 220 °C for 1 hour and quenched in water at room temperature. Then a 24-hour re-aging process was performed at 120 °C.

The annealed and RRA heat treated AA7075 samples were tensile tested at room temperature and at 100, 200, and 300 °C. Tensile tests at room temperature and elevated temperatures were performed at a strain rate of 0.001/s using a Zwick/Roell Z600 mechanical tester, which has a load capacity of 600 kN and can perform tensile testing up to 1100 °C. The tests were repeated for each sample to ensure experimental accuracy. The gage length of the cylindrical samples was 35 mm and the inner diameter of narrow section was 6 mm.

Samples were taken from the tensile tested specimens (from undeformed regions) and characterization studies were performed. ASTM E3-11 standard was used for metallographic examinations. Etching process was performed with Keller solution for

45- 60 seconds. Carl Zeiss Ultra Plus Gemini SEM scanning electron microscope (SEM) equipped with a Bruker X Flash 6/10 energy distribution spectrometer (EDS) was used for microstructure viewing analysis. In addition, X-ray diffraction (XRD) analysis was performed at a rate of 3 degrees/s in the range of 10-90 degrees using a Rigaku Ultima IV XRD analyzer to determine the changes and secondary phases in the microstructure. Hardness measurements were performed using a Shimadzu micro-hardness device (HV0.5, 500gf for 15 seconds). Each hardness measurement was repeated five times and mean values were calculated. Values such as yield stress, maximum tensile stress, modulus of elasticity, and percent elongation were calculated from the curves obtained from tensile tests.

RESULTS AND DISCUSSIONS

Microstructural Investigation

Figure 1 shows the microstructure SEM images of the AA7075 alloy samples tensile tested at temperatures between 100 °C and 300 °C. As seen in the SEM images, the secondary phase precipitates formed in the structure both at the grain boundaries and within grains. This can be interpreted as the dispersion of Al-Cu-Mg eutectic or Al-Cu eutectic phases by increasing the solubility of the matrix phase because of high temperature [21]. It appears that the microstructures of annealed and RRA-applied samples were similar with each other. Comparing the microstructure SEM images given in Figure 2, it can be observed that secondary phase precipitates in the RRA heat-treated alloy were generally located at grain boundaries. This may be because of the

precipitates formed in RRA samples were denser in the structure. It was also noted in some previous studies that heat treatment methods and parameters effect the size and volume ratios of secondary phase precipitates [6,12]. Looking at the EDS results in Table 2, it is understood that points 2, 5, 11, and 14 are Si-rich zones. Figure 2 shows the results of the XRD analysis performed after the tensile tests. The XRD results given in Figure 2 indicate that matrix phase (PDF Card No: 00-001-1176) as well as Al₂Cu (PDF Card No: 00-002-1309) and MgZn₂ (PDF Card No: 00-034-0457) phases formed in the structure of the alloys. It was also noted in some previous studies that the MgZn₂ phase, expressed as stable and incoherent η' , is present in the structure of AA7075 alloys [11,17,22].

Hardness Results

Figure 3 shows the hardness of the annealed and RRA heat treated AA7075 alloys which was tensile tested at room temperature and elevated temperature. The hardness values found for the annealed and RRA heat treated AA7075 alloy samples at room temperature were 76.85 (± 2.51) and 161.75 (± 2.165) HV, respectively. It seems that the RRA heat treatment improved the hardness. The hardness value found for the RRA heat treated AA7075 alloy sample was approximately 2 times the hardness value of the annealed sample. Aging heat treatments are known to increase the hardness of the AA7075 alloy significantly. The hardness results found in this study are similar to those obtained in some previous studies [4,22,23,24]. It is known that the hardness of the AA7075 alloy increases with the formation of second phase precipitates in the matrix as a result of aging heat treatments [25]. In general, the strengthening mechanism can be

explained as obstructing the dislocation movement by secondary phase particles in the matrix and thus increasing the stress level required for deformation. In a previous study, it was noted that precipitates in T6 heat treated alloys show intense and continuous distribution at grain boundaries [7]. The size of these secondary phase precipitates increases with re-ageing, resulting in a slight decrease in hardness [26]. It was observed that there was a decrease in the hardness of tensile tested samples with increasing temperature. When the test temperature increased from 100 °C to 300 °C, the hardness of the annealed alloy dropped from 72.725 (± 1.158) HV to 65.766 (± 1.184) HV. Similarly, for the RRA heat treated samples, the hardness decreased from 156.25 (± 3.112) HV to 132.25 (± 2.277) HV. This is due to grain coarsening caused by over-aging in the material due to increased temperature [12]. As can be seen from the hardness results, the RRA heat treated samples exhibited better hardness behavior than the annealed samples at 300 °C.

Tensile Test Results

Figure 4 shows the stress-strain curves obtained from the tensile tests of the annealed and RRA heat treated AA7075 alloys performed at room temperature and elevated temperatures. It was observed that the peak stress value of the RRA heat treated AA7075 alloy increased, while the ductility decreased. The stress-strain curves of the annealed and RRA heat treated AA7075 alloys tended to decrease with increasing temperature. This indicates that deformation occurred at lower stress levels with increased temperature. In other words, the strength decreased as the temperature increased. In addition, the elongation values of the samples increased with the

increasing temperature. This increase in ductility is an indication that grain boundary sliding, which is a deformation mechanism, was also activated at elevated temperatures [27]. Figure 5 shows the values such as yield stress and tensile stress, calculated based on the stress-strain graphs. As seen in Figure 5, the yield strength and tensile strength of the annealed samples at room temperature was 183.536 and 274.245 MPa, respectively. The RRA heat treated AA7075 alloy samples were found have a yield strength of 493.268 MPa and a tensile strength of 548.564 MPa at room temperature. From these results, it can be clear that a significant increase in strength was achieved by RRA heat treatment. The elongation of the AA7075 alloy annealed at room temperature decreased from 19.219 to 12.771 by RRA heat treatment. This decrease in ductility with increased strength may be because of stable incoherent secondary phases became coherent with the matrix by the influence of high temperature [19]. Both the annealed and RRA heat treated samples exhibited a decrease in strength and an increase in ductility with the increasing temperature. Compared to the characteristics of the samples tensile tested at room temperature and 300 °C (Figure 5), there was an increase in elongation by approximately 134% in the annealed samples and 18% in the RRA heat treated samples. It was observed that the ductility increase in the annealed samples was much higher than the RRA treated samples. When analyzing the yield stress and tensile stress results, it seems that there was a 42% decrease in the yield stress and a 62% decrease in the tensile stress of the annealed samples and a 55% decrease in the yield and tensile stress of the RRA heat treated samples with increased temperature. The RRA treated samples were observed to show almost similar mechanical properties at

elevated temperatures with the mechanical properties of annealed samples at room temperature. Mechanical properties of metals are known to decrease with increasing temperature. For aluminum, steel, tungsten and MgO, the modulus of elasticity value was reported to decrease with respect to the homologous temperature. The homologous temperature T/T_m refers to the ratio of ambient temperature to melting temperature. A significant decrease in the strength with increasing temperature is observed in many materials around a value of 0.5 of the homologous temperature [28]. In terms of mechanical properties, the RRA heat treated AA7075 alloy samples exhibited a negligible decrease in strength. This can be attributed to the coarser and higher amount of secondary phase precipitates formed in the RRA heat treated AA7075 alloy samples due to longer heat treatment exposure by retrogression and re-aging. A similar case is expressed as the increase in the of the size of secondary phases in the structure at long aging times [14]. Figure 6 shows the elongation and cross-section shrinkage of the AA7075 alloys. Looking at the elongation at the fracture, the annealed samples exhibited greater elongation up to 300 °C compared to the RRA heat treated samples.

Fracture surfaces of the annealed and RRA heat treated AA7075 alloy samples were examined. Figure 7 shows the SEM images of these surfaces. It seems that the neck diameter of the samples tensile tested at 300 °C was about 85% of the annealed samples tensile tested at room temperature and about 65% of the RRA heat treated samples.

Dimples were observed to have formed on the fracture surfaces of the samples tensile tested at room temperature and elevated temperatures, and a ductile fracture occurred. These dimples formed on surfaces are caused by cavities in the microstructure or by second phase precipitates. A previous study reported that second phase precipitates in aging aluminum alloys cause dimple formation on fracture surfaces and exhibit ductile behavior [29]. Additionally, the formation of these dimples on fracture surfaces also contributes significantly to the strain loading and the coalescence of micro-voids during tensile tests [30]. These micro-voids can also be caused by particle (precipitate) matrix decomposition or particle breaking caused by stress during tensile testing [31]. Also, it seems that the fracture surface SEM images are close to those obtained in similar studies in the literature [30,32–34]. It was observed that more regular, coarse, and larger dimples formed with increasing temperature. Since the alloy elongates more at high temperatures, the voids in the microstructure coalesce and become larger and appear as larger dimples on fracture surfaces. The reason behind the RRA heat treated alloy containing larger dimples than the annealed alloy may be the precipitate growth caused by over-aging of secondary phase precipitates in the microstructure.

CONCLUSIONS

In this study, mechanical properties of annealed and RRA heat treated AA7075 alloys were examined via tensile tests at elevated temperatures up to 300°C. The characterization analysis and hardness investigations were conducted at room temperature after the samples tensile tested at elevated temperatures. As a result of

the investigations with scanning electron microscopy, it was observed that secondary phase precipitates formed in the structure by RRA heat treatment. The RRA heat treated samples showed better mechanical properties even at elevated temperatures compared to the mechanical properties of the annealed sample at room temperature. The increasing test temperature led to a decrease in all mechanical properties. A significant decrease was observed in mechanical properties at 300 °C. It was found that the decrease in mechanical properties with increasing temperature was more pronounced for the RRA heat treated samples than the annealed samples. The annealed AA7075 alloy exhibited more ductile behavior than the RRA heat treated alloy. An increase in ductility was observed in parallel with the increasing temperature. The increase in ductility with increasing temperature was found to be higher in the annealed samples (compared to the RRA heat treated samples). In fracture surface examinations, it was found that the samples were deformed by a ductile fracture mechanism at room temperature and elevated temperatures.

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Figure Captions List

- Fig. 1 Microstructure SEM images of AA7075-O and AA7075-RRA samples tensile tested at different temperatures.
- Fig. 2 The XRD results of the AA7075-O and AA7075-RRA samples: a) AA7075-O, b) AA7075-RRA.
- Fig. 3 Hardness changes of the annealed and RRA heat treated AA7075 alloy samples at different temperatures.
- Fig. 4 The stress-strain curves of the annealed (a) and RRA heat treated (b) AA7075 alloy samples at different temperatures.
- Fig. 5 Mechanical properties of the AA7075 alloy samples treated with different heat treatments at different temperatures; tensile stress (a), yield stress (b)
- Fig. 6 Percent elongation and cross-section shrinkage of the AA7075 alloy samples at the time of fracture at different temperatures
- Fig. 7 Fracture surface SEM images of the annealed and RRA heat treated AA7075 samples tensile tested at different temperatures.

Table Caption List

Table 1 Chemical composition of the AA7075 alloy

Table 2 The EDS results of annealed and RRA heat treated AA7075 alloys

Table 1.

Al	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Oth.
90.05	0.21	0.2	1.52	0.11	2.28	0.2	5.27	0.05	0.11

Table 2.

Point	Al	Mg	Si	Mn	Fe	Cu	Zn
1	91.64	1.62	0.1	0.29	0	2.15	4.2
2	50.16	1.39	38.99	0	0.53	5.87	3.06
3	13.17	0.93	0.33	0.03	0.43	77.02	8.09
4	85.8	2.88	0	0.86	0	2.74	7.73
5	36.2	1.68	55.04	0.83	0	2.17	4.09
6	59.65	1.38	0.55	0	1.05	31.94	5.42
7	68.91	2.45	0	0.05	0	21.59	7.01
8	72.73	1.49	0.1	0.95	1.25	19.1	4.37
9	86.1	2.43	0	0.17	0.67	4.2	6.43
10	85.55	2.75	0.23	0.26	0.04	3.46	7.71
11	53.67	1	37.98	0.07	0	4.97	2.3
12	42.09	0.86	0.07	0	0.59	52.47	3.91
13	90.36	1.58	0	1.03	1.51	1.47	4.04
14	64.51	2.32	25.05	0	0.19	2.09	5.84
15	85.27	2.54	0	0.87	0	3.43	7.89

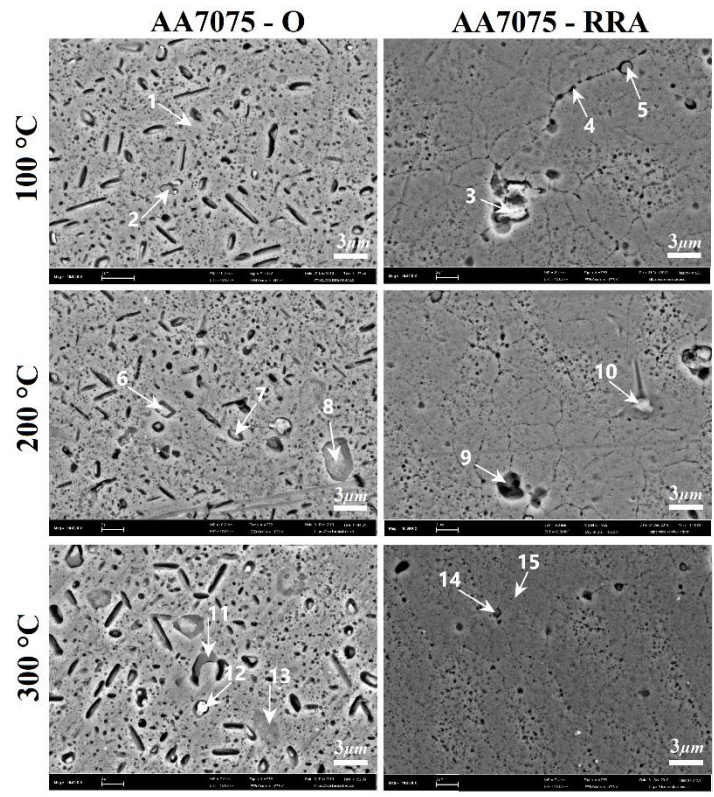


Figure 1.

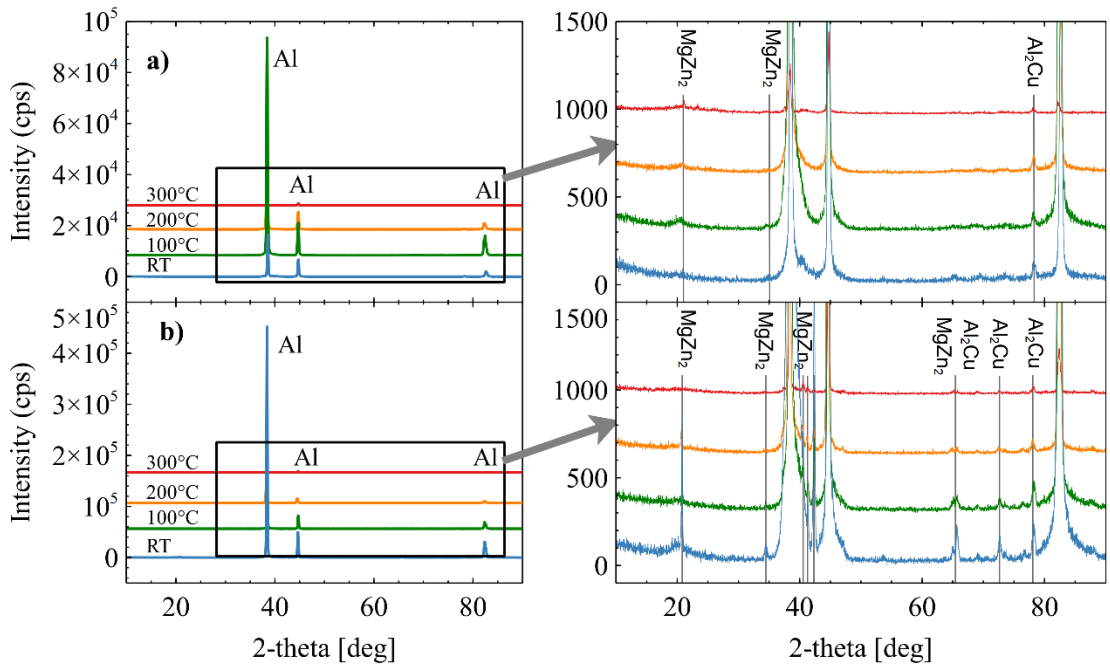


Figure 2.

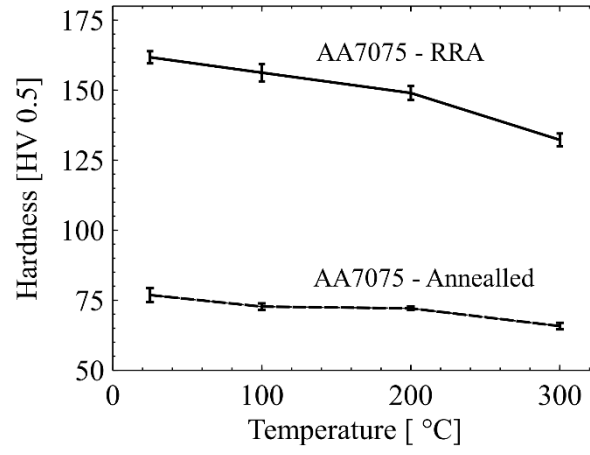


Figure 3.

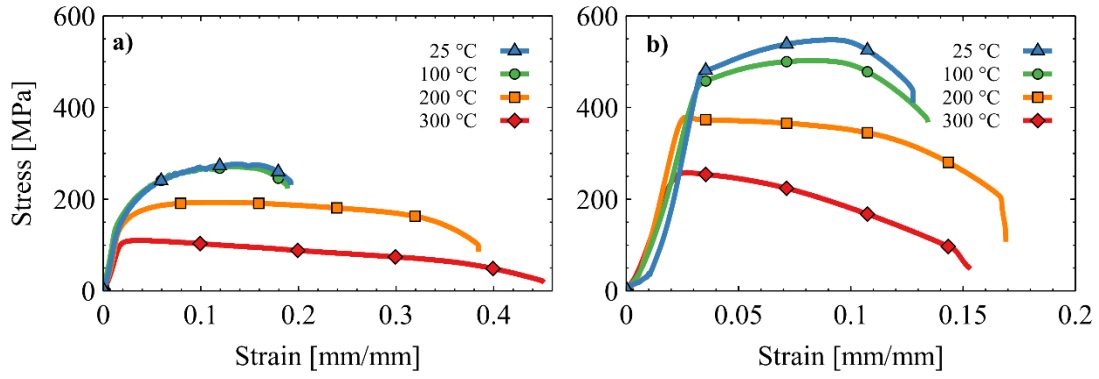


Figure 4.

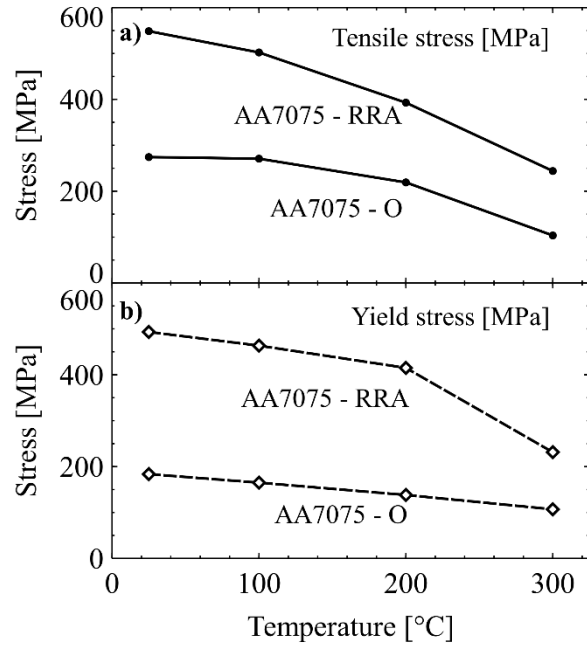


Figure 5.

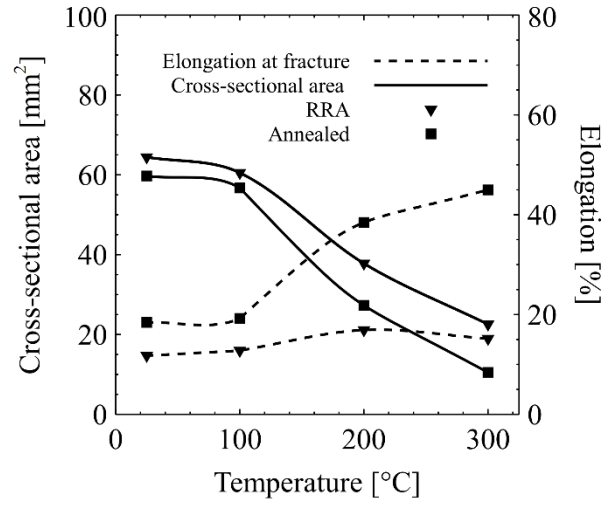


Figure 6.

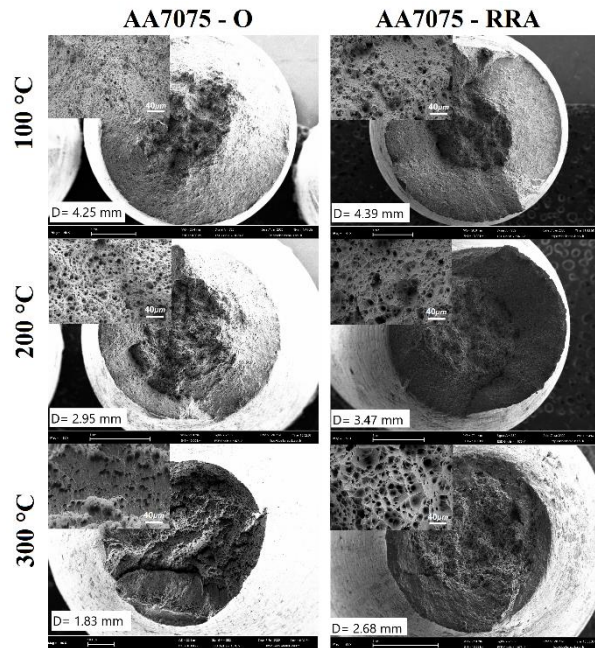


Figure 7.