High-temperature compressive behavior and kinetics analysis of Al$_{0.4}$MnCrCoFeNi high entropy alloy

Hamed Kaypour$^1$, Said Nategh$^1$, Reza Gholamipour$^2$ and Alireza Khodabandeh$^1$

$^1$ Department of Materials Engineering, Science and Research Branch, Islamic Azad University, Tehran, Iran
$^2$ Iranian Research Organization for Science and Technology (IROST), PO Box 33535111, Tehran, Iran

E-mail: rgholamipour@gmail.com

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Abstract

In the present study, Al$_x$MnCrCoFeNi alloys ($x = 0, 0.4$) were produced by vacuum melting and casting. For this aim, ingots were subjected to the homogenization, cold roll, and annealing. X-ray diffraction analysis and SEM images indicated that the crystal structure is FCC solid solution for MnCrCoFeNi. Moreover, in addition to FCC solid solution, the alloy with aluminum ($x = 0.4$) ordered B2 precipitates. Besides, hot compressive tests were conducted at different temperatures and strain rates of 0.01, 0.05 and 0.1/s. Investigating the stress-strain diagrams and work hardening rate, it was revealed that twins played a role in the deformation mechanism. In the constitutive equation, the values of the stress exponent and activation energy were measured and they were 6.86 and $434 \text{ kJ mol}^{-1}$, respectively. The exponential equations of peak stress and peak strain, as well as steady-state, were obtained due to the Zener-Hollomon parameter. Regarding the linear relationship between the work hardening rate and the dislocation annihilation coefficients, the activation energy of deformation changes at 600 °C. Finally, another effect of the Al addition was shown to be increase in initiation temperature of necklace structure about 200 °C in hot compression test. In addition, B2 nano precipitate and nanotwins indicated that there was twin deformation mechanism in alloy containing Al.

1. Introduction

High entropy alloys are new materials which contain at least 5 major elements with the concentrations of 5–35 at.% [1] equimolar ratios with a configuration entropy greater than or equal to 1.6R (R: gas constant) [2]. Recent studies have shown that high entropy alloys have excellent properties such as wear resistance [3], high hardness [4], low temperature strength [5], low temperature resistance [6], oxidation resistance [7], compressive strength [8], refractory properties [9] and superplasticity [10]. In these alloys, the configurational entropy can exceed the enthalpy of compound formation, which makes random solid solution formation [11]. The main structure, despite its various elements, is often BCC, FCC, or a mixture of both. With equimolar ratios, MnCrCoFeNi has a single-phase face center cubic which has been discovered by Cantor et al [12]. A number of studies have recently revealed that the mechanical properties of single-phase high entropy alloys at room and elevated temperatures are often not sufficient for the engineering applications. One of the effective methods for strengthening alloys is intentional addition of intermetallic as second phases. Intermetallic compounds can have significant effects on the improvement of mechanical properties [13, 14]. For example, Al is one of them for this purpose, which can increase the mechanical properties due to the effect of lattice distortion [15] and formation of intermetallic compounds [16]. Due to large enthalpy of mixing ($\approx 22 \text{ kJ mol}^{-1}$), there is a strong trend to form intermetallic compounds with nickel. Ni-Al Rich phase with BCC crystal structure (B2) is often observed in high entropy alloys containing these two elements [17, 18]. The addition of Al into MnCrCoFeNi destabilizes the crystal structure from FCC to FCC + BCC and finally a harder BCC phase is formed [19]. This phase transformation can be attributed to low valence electron concentration of Al compared to other elements [20].
Several studies have been conducted on high entropy alloys containing Al\(^{21–27}\), but a limited number of studies have so far been performed on the mechanical properties of Cantor alloys containing Al, especially at high temperatures. Some of these studies have investigated the effects of Al addition on tensile properties of the FeCoNiCrMn\(^{15}\), phase stability of CrMnFeCoNiAl\(^{0.25}\)\(^{28}\), solid solution strengthening of CoCrFeNiMnAl\(^{x}\)\(^{29}\), formability of Al\(^{0.5}\)CoCrFeMn\(^{i}\)\(^{30}\) and strength at medium temperature CrMnFeCoNiAl\(^{0.75}\)\(^{31}\). In the present study, however, the compression tests were carried out at different processes to elucidate precipitation behavior and hot deformation mechanism. In this study, the Al effect on microstructure, work hardening rate, kinetic equations, and activation energy of hot deformation of Al\(^{0.4}\)MnCrCoFeNi alloy have been investigated.

2. Materials and method

2.1. Samples preparation

The elements of Al, Cr, Co, Fe, Mn, and Ni with more than 99.5% purity were used as raw materials. MnCrCoFeNi and Al\(^{0.4}\)MnCrCoFeNi alloys were produced using vacuum arc melting with a water-cooled copper crucible (Vacuum: 10\(^{-5}\) torr, Capacity: 500 gr, Argon purity: 99.9995%, Argon pressure: 0.5bar, Electrode: Tungsten). An additional 3.0 at.% Mn was added to compensate for its evaporation during melting. Moreover, Manganese oxide was removed by an aqueous solution of nitric acid. Alloys were remelted for at least 5 times to improve chemical homogeneity. The dimensions of ingots were about 12 mm \(\times\) 25 mm. For brevity, the alloys of Al\(^{0.4}\)MnCrCoFeNi and MnCrCoFeNi have been shown as Al\(^{0.4}\) and Al\(^{0}\), respectively. The chemical analysis of samples (EDX) are given in table 1.

| Table 1. Chemical composition of Al\(^{0}\) and Al\(^{0.4}\) alloys (at.%). |
|-----------------|---|---|---|---|---|---|
|                | Cr | Mn | Fe | Co | Ni | Al |
| Al\(^{0}\)      | 19.85 | 19.67 | 20.26 | 20.01 | 20.21 | 0 |
| Al\(^{0.4}\)    | 18.56 | 18.64 | 18.44 | 18.58 | 18.19 | 7.59 |

2.2. Heat treatment

Ingots were homogenized at 1200 °C for 7 h to remove segregation during solidification and then 55% cold-rolled reduction. Subsequently, the cold-rolled samples were annealed at 1100 °C for 1 h followed by water quenching (Furnace Model: ALF18, Company: Atbin, Temperature Stability: ±1 °C, Max Power: 4 kW, Max Temp: 1200 °C, Vol: 18 lit).

2.3. Mechanical tests

Compression tests were performed on cylindrical specimens with the dimensions of 6 mm \(\times\) 9 mm using Instron 8503 machine at room and high temperatures with strain rates of 0.01, 0.05, and 0.1 /s.

2.4. Microstructural characterization

Microstructural analysis was performed on deformed samples which were sectioned parallel to the compression direction. Samples were cut, mounted, ground, polished to 5000 grit and then with 0.3 micron alumina suspension, and cleaned by ultrasonic and etched with aqua regia solution. Microstructural characterization was performed by an optical microscope using an Olympus BX51M equipped with DP26 digital camera and field emission scanning electron microscopy (FE-SEM MIRA3 TESCAN), equipped with energy dispersive spectroscopy (EDS). A transmission electron microscope (TEM) (FEI Tecnai G\(^{2}\) F20) with an integrated energy dispersive X-ray spectroscopy system was used to obtain the crystallographic information. The samples for TEM were prepared using a standard technique, including cutting, grinding, dimpling and jet polishing in an electrolyte solution containing 95% ethanol and 5% perchloric acid at 12 °C and 30 V. In addition, XRD analysis was performed using a Philips PW1730 with Cu K\(_{\alpha}\) radiation (\(\lambda = 1.54060\) Å), while the diffraction angle ranged from 20° to 100°, with step size of 0.02°/s. The voltage and current were 40 kV and 30 mA, respectively.

3. Results and discussion

3.1. Microstructure characterization

Figure 1 shows the XRD pattern of Al\(^{0}\) alloy. The results revealed that the microstructure consisted of FCC crystal structure. XRD pattern of Al\(^{0.4}\) alloy shows that, besides FCC crystal structure, has several peaks in the 43 and 81° angle ranges which correspond to AlNi-rich phase with B2/BCC crystal structure. Comparing XRD
patterns of A10 and A0.4, it can be seen that with the addition of Al, the peaks shifted to lower angles. The atomic radius of Al was larger than other alloy elements such as Ni, Co, Fe, Mn, and Cr\[32]. Therefore, the distance between the atomic planes increased. Hence, due to Bragg’s law, it could be concluded that with Al addition, the lattice parameter had become larger, and distortion was increased.

Figures 2(a) and (b) show the SEM images of A10 and A0.4 alloys, respectively. Precipitated particles are only seen in the matrix in A0.4 alloy. Therefore, it seems that the formation of twins in A0.4 and A0 alloy is inevitable. According to the literature, in alloys with small stacking-fault energies like MnCrCoFeNi, twins are the main deformation mechanism. As Mishra et al\[33, 34] showed, lattice strain had an important effect on the dislocation core energy and consequently on the formation of stacking-fault and its energy. The lattice strain in high-entropy alloys increased the crystal energy, reducing the energy required for nucleation and the formation of dislocations and twins\[35]. The lattice strain calculated by Williamson–Hall method\[36–38]\ revealed that the addition of Al to Contor alloy increased the lattice strain by 62.5% (0.0024 and 0.0039 for A10 and A0.4, respectively).

Another important relevant parameter was atomic size difference:

\[
\delta = \frac{1}{n} \sum_{i=1}^{i=n} c_i \left( 1 - \frac{\eta_i}{\sum_{i=1}^{i=n} c_i \eta_i} \right)^2
\]

where \(c\) is the molar percent, \(r\) is the atomic radius and \(n\) is the number of alloy components\[39]. Moreover, these calculations indicated that the addition of Al to Contor alloy significantly increased the atomic size difference parameter by 2.3 times (Atomic size difference for A10 and A0.4 alloy is 1.12 and 3.7, respectively). Therefore, investigation of these two parameters revealed that the addition of Al reduced the stacking-fault energy. The results of Kumar et al are in line with the present result\[34].

Figure 1. XRD patterns for A10 and A0.4 alloys.
The X-ray elemental map of Al0.4 were used to characterize the precipitates, which have been shown in figure 3. It shows that precipitates have been enriched in Al and Ni and also contain Mn and Co with uniform distribution; small amounts of Cr and Fe elements. XRD results confirmed that precipitates were Al-Ni-Rich B2 phase (figure 1).

The metallographic images of the compressive test specimens of Al0 and Al0.4 alloy are shown in figure 4. Images in this figure are taken from the cross sections in which the stress-direction is perpendicular to the scale bar. Figures 4(a) and (b) show the metallographic images of the compressive test specimen of Al0 alloy at 25 °C and 800 °C in which dynamic recrystallization has occurred, and the necklace structure is evident at the grain boundaries, and figure 4(c) illustrates Al0 SEM image of necklace structure. However, to better assess the temperature at which the necklace structure is formed, figure 4(d) shows the metallographic image of a compressive test specimen of Al0.4 alloy at 800 °C, and it is clear that no trace of necklace structure can be observed, while by increasing the test temperature to 1000 °C in figure 4(e), the necklace structure is formed. Figure 4(f) also shows the SEM image of the compressive test of the same specimen at 1000 °C. The presence of B2 precipitates, which also have high thermal stability, blocked the movement of grain boundaries. Therefore, Al presence has increased the recrystallization temperature and delaying of softening, leading to conserving the strength at high temperatures [40].

3.2. Work hardening rate
The flow curves of Al0 alloy at different temperatures with a strain rate of 0.01/s are represented in figure 5(a). As it can be seen, the flow curves gradually decrease with increasing temperature and reach steady-state stress with decreasing slope at 600 °C and 800 °C. Steady state stresses at 600 °C and 800 °C are 480 and 298 MPa, respectively, and two curves at 25 °C and 400 °C still show hardening behavior. The flow curves of Al0.4 alloy at different temperatures are shown in figure 5(d). In this diagram, the peak stress of 280 MPa has been observed at a strain of 0.15 and a temperature of 800 °C. However, with the increase in temperature from 400 °C to 600 °C, there is no noticeable decrease in the strength and the alloy also shows work hardening behavior, except for the temperature of 800 °C.

The effect of temperature on the work hardening rate ($\theta = \frac{d\sigma}{d\varepsilon}$) due to plastic strain for Al0 is shown in figure 5(b). Many changes in the work hardening rate are observed with the continuation of deformation process. After an initial change in the slope of curve at all temperatures, a linear region is seen, and then the slope value reaches zero. At 25 and 400 °C, the slope of curve is negative in all the stages so that, after an initial change, 0.08 strain linear region is started. However, at higher temperatures, the sample’s behavior has changed so that at a temperature of 600 °C, after a steep and negative slope in the plastic strain 0.02, the slope becomes positive, and then in the strain 0.08, the linear region starts. The same is true for the samples tested at 800 °C, except that the slope decrease occurs at 0.034 plastic strain, and the linear region starts at 0.059 strain. The sharp decrease in the work hardening rate in the early stages may be due to the recovery process that occurs by the cross slip of the dislocations and the annihilation of dislocations with the opposite sign [41]. Figure 5(e) shows the effect of temperature on the work hardening rate due to plastic strain for Al0.4. In all the curves, a positive slope is
observed, the smallest of which is related to the temperature of 800 °C and the largest of which is related to 600 °C. The linear region starts at ambient temperature and 400 °C at a strain of 0.08 and 0.03, respectively. By an increase in temperature to 600 °C, the slope has become positive at 0.03 strain, and the linear region has started at 0.07 strain. The positive slope which can be caused by the formation of twins, is quite noticeable at this temperature. By an increase in temperature to 800 °C, the graph slope has sharply increased. Due to the diagrams, it is observed that with increasing temperature from 400 °C to 600 °C, there is no noticeable decrease in strength, which is probably due to the formation of twins at this temperature.

The work hardening rate of Al0 due to flow stress is shown in figure 5(c), which is similar to that in figure 5(b). The dashed line in figure 5(c) represents the value of $\theta = \sigma$. The region below this line ($\sigma > \theta$) is the non-uniform plastic deformation zone. Increasing the temperature causes the curve to shift to lower stresses. Also, the linear regions which are proportional to dislocation annihilation [42], are almost parallel to each other, except for the temperature of 800 °C (this will be discussed in 3.4 section). At first, at low stresses, the work hardening rate decreases sharply, and then at high stresses, a gradual decrease is observed. Figure 5(f) shows the work hardening rate of Al0.4 due to flow stress. The presence of a peak is seen in the diagrams so that the largest peak is observed at 600 °C (as in figure 5(e)). The increase in work hardening rate is due to the low stacking fault energy and the twinning mechanism activation during the alloy deformation [43]. In solid solution and high-entropy alloys, due to high lattice strain energy, the stacking-fault energy is low [44], and in these types of alloys, twin boundaries act as production sources of dislocation and are also considered as a barrier to its movement, leading to increase of the dislocation density [45, 46].
One more important factor which has increased the strength and work hardening rate of Al0.4 alloy is the presence of B2 precipitates, which acts as a barrier against dislocation movement. In other words, twin boundaries and precipitates increase the dislocation density as one of the important factors in increasing the work hardening rate [47–49]. A new strategy to increase the strength is creating nanostructures such as nanotwins and nanophases, which decreases the mean free path of dislocations. High-temperature stable nanophase increases the thermal stability of nanotwins resulting in a good combination of ductility and strength [50].

Twin boundaries can both act as dislocation sources and efficient barriers to dislocation motion; therefore they pose an important impact on strengthening of alloys. In nanotwinned structures (NT), dislocation-twin boundary interactions are different from dislocation-dislocation and dislocation-grain boundary interactions. Due to these interreactions, NT materials show unique mechanical properties and any change in twin boundaries is highly contributive to strengthening effect of NT materials. Hence, a new kind of NT materials called Hierarchical nanotwined (HNT) have been developed. HNT alloys show high strength, good ductility, and proper strain hardening ability. HNT structures can improve a material strength without sacrificing its ductility [51, 52]. According to the TEM images in figure 6, the twin boundaries of primary twins (T1) act as dislocation sources as well as barriers to secondary twins (T2), which can increase the strength.

Figure 6(a) shows the twins and high-density dislocations of the deformed sample at room temperature. The corresponding selected area electron diffraction pattern of FCC twins at [011] orientation is also shown in the inset indexing of two principal reflections. Figure 6(b) shows the micrograph of the deformed sample at 600 °C. Three principal reflections of the FCC matrix have been indexed. High-resolution TEM images of nanotwins with their corresponding Fast Fourier Transformed (FFT) diffraction patterns are shown in figures 6(c) and (d).
The nanotwins have a mean thickness of 20 nm with a sharp wall in room temperature specimen (figure 6(c)); however, by increasing the deformation temperature to 600 °C, the sharpness decreases (figure 6(d)).

Figure 7(a) indicates B2 precipitate and its SAED of the sample deformed at room temperature. Three principal reflections and superlattice reflection (100) have been indexed. Figure 7(b) also reveals B2 precipitate with a high dislocation density of a deformed sample at 600 °C. Figures 7(c) and (d) show the relatively low magnification bright field micrograph of B2 precipitates in Al0.4 Alloy deformed at 25 °C and 600 °C, respectively. FCC twins along [011] zone are indexed.

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Figure 7(a) indicates B2 precipitate and its SAED of the sample deformed at room temperature. Three principal reflections and superlattice reflection (100) have been indexed. Figure 7(b) also reveals B2 precipitate with a high dislocation density of a deformed sample at 600 °C. Figures 7(c) and (d) show the relatively low magnification bright field micrograph of B2 precipitates in Al0.4 Alloy deformed at room temperature and 600 °C, respectively. Due to the presence of nanoprecipitates of B2 phase in the structure, which increase the stability of nanotwins and the high density of nanotwins, it seems that the dominant deformation mechanism at high temperatures is twinning. Also it is evident that the HNT structure plays an important role in maintaining the strength at high temperatures.

3.3. Compressive strength and Hollomon analysis
The effect of temperature on Yield Strength (YS) and ultimate compressive strength (UCS) at 0.01/s strain rate are shown in figure 8. By increasing temperature, there is a continuous decrease in the YS and UCS of Al0 alloy, while in Al0.4 alloy, the YS at high temperature is higher than Al0 alloy, and the decrease in strength with temperature has a slight gradient. It is possible that the B2 phase, especially in the form of nano precipitate, is responsible for the high-temperature strength of Al0.4 alloy because the B2 phase is stable at high temperatures [53].

The relationship between stress and strain is defined due to Hollomon’s equation, as follows [54]:

\[ \sigma = K \varepsilon^n \]  

where \( \sigma \) is the stress, \( \varepsilon \) is the plastic strain, \( K \) is the constant, and \( n \) is the work hardening exponent. Work hardening exponents are 0.23, 0.21, 0.19, 0.18 for Al0 and 0.23, 0.26, 0.27 and 0.10 for Al0.4 at 25, 400 °C, 600 °C and 800 °C, respectively. In the obtained linear equations, the linear regression coefficient is higher than 0.97. The effect of temperature on work hardening exponents is shown in figure 9.

As it is evident, the work hardening exponent of Al0 alloy decreases by increasing the temperature from 0.23 at ambient temperature to 0.18 at 800 °C. But in the Al0.4, the work hardening exponent not only does not
decrease with increasing temperature but also increased up to 600 °C, indicating that the alloy with a higher work hardening exponent has higher strength and ductility [54]. High-entropy alloys have a high angle grain boundary due to severe distortion and high entropy. Therefore, these alloys are less stable and the changes are high in their mechanical properties with temperature during the grain boundary relaxation process, and reducing the system energy by these high-energy boundaries is easier than the conventional systems [55]. Increasing the work hardening exponent with a temperature up to 600 °C which is related to strength and even hardness (not presented in this report), can be related to the stability of grain boundaries during the grain boundary relaxation process due to the high temperature [55]. It seems that increasing the temperature up to 800 °C has provided the necessary conditions for the dislocation to cross the obstacles and, as a result, the
dislocation has acted as the dominant mechanism in the plastic deformation [56]. The steep slope of work hardening rate in figure 5(e) expresses this issue.

3.4. Kinetics analysis
Numerous experimental equations were proposed to calculate the deformation activation energy of the alloys. The following equations are among the most common equations which describe the behavior of alloys in different deformation conditions. These equations show the effect of strain rate and temperature on different stresses. Zener-Hollomon parameter determines the relationship between these parameters according to the following equations:

\[ A' \sigma_p^{\alpha} = \dot{\varepsilon} \exp \left( \frac{Q}{RT} \right) = Z \]  
(3)

\[ A' \exp(\beta \sigma_p) = \dot{\varepsilon} \exp \left( \frac{Q}{RT} \right) = Z \]  
(4)

\[ A(\sinh (\alpha \sigma_p))^n = \dot{\varepsilon} \exp \left( \frac{Q}{RT} \right) = Z \]  
(5)

where, \( A, A', A'', \alpha, \beta, n, n' \) are constant and Q is the activation energy associated to the deformation, R is the gas constant, Z is the Zener-Hollomon parameter, and \( \sigma_p \) is the peak stress [40].

Figure 10 shows the true stress-strain diagram of Al0.4 alloy at different temperatures and different strain rates. It seems to be the peak stress in all diagrams, some of which are accompanied by steady-state stress. According to the diagrams, by increasing strain rate, the peak stress \( \sigma_p \), peak strain \( \varepsilon_p \), and also steady-state stress
\( \sigma \)

increase. On the other hand, it can be observed that if the strain rate is constant, by decreasing temperature due to slowing down the recovery process, peak stress, peak strain, and steady-state stress increase. The information obtained from these diagrams under different temperature conditions and strain rates is given in Table 2. Peak stress (\( \sigma_p \)) and peak strain (\( \varepsilon_p \)) can be calculated from the \( \theta - \sigma \) diagram where \( \theta = 0 \).

Taking the logarithm from both sides of equation (3):

\[
\ln \sigma_p = 1/N' \ln \dot{\varepsilon} + Q/RT - \ln A'
\]

(6)

Due to equation (5), at a constant temperature, the relationship between peak stress and strain rate (Figure 11(a)) is linear at the logarithmic scale, and the line slope shows \( N' \) value. Moreover, if the logarithm is taken from both sides of equation (4), at the constant temperature, a linear relationship is observed between the peak stress and the logarithm of strain rate (equation (7)) and from the slope of the diagram (Figure 11(b)) the parameter \( \beta = 0.0294 \) is obtained. Therefore, the value of \( \alpha = 0.0031 \) can be calculated.

\[
\beta \sigma_p = \ln \dot{\varepsilon} + Q/RT - \ln A'
\]

(7)

Also, according to equation (5) and Figure 11(c), there is a linear relationship between the logarithm of strain rate and the logarithm of hyperbolic sine at 700 °C and 800 °C, where the average value of \( n \) is 6.45 due to the slope of the line. Similarly, if the logarithm of the hyperbolic sine is plotted at different temperatures at a constant strain rate (Figure 11(d)), a linear relationship with a regression coefficient close to one can be observed. Regarding the slope of graph which is equal to \( Q/nR \), the amount of activation energy (\( Q \)) is 434 kJ mol\(^{-1}\).

Table 3 shows the activation energy for some steels and high entropy alloys. As it is shown, the activation energy of high-entropy alloy is much higher than that in other steels. The reported maximum activation energy of MnCrCoFeNi (Al0) alloy is 350 kJ mol\(^{-1}\) [37], which increases to 434 kJ mol\(^{-1}\) with the addition of Al (Al0.4). The high activation energy of Al0.4 alloy is related to the formation of precipitates (pinning effect) and solute drag effects [58]. A similar phenomenon has been observed with the addition of Al in High Mn TWIP steel [59].

Equation (5) shows the relationship between peak stress, strain rate, and temperature in the hot deformation process. Sellar and Tegart first proposed this equation [60]. By taking the logarithm from the sides:

\[
\ln Z = \ln A + n \ln [\sinh (\alpha \sigma_p)]
\]

(8)

The values of A and n can be obtained by plotting \( \ln [\sinh (\alpha \sigma_p)] \) due to \( \ln Z \), calculating the slope of the line, and the point of intersection with the vertical axis. The points drawn in Figure 12 indicate the linear relationship with the regression coefficient close to one.

The values of \( n = 6.86 \) and \( \ln A = 44.07 \) were calculated. Therefore, the constitutive equation can be written as follows:

\[
\dot{\varepsilon} = A (\sinh (\alpha \sigma_p))^{n} \exp \left( -\frac{Q}{RT} \right)
\]

(9)

\[
\dot{\varepsilon} = [\sinh (0.0031 \sigma_p)]^{6.86} \exp \left( 44.07 - \frac{52202.4}{T} \right)
\]

(10)

The exponential equation for peak stress and also strain as a function of the Zener–Hollomon parameter can also be written as follows [63]:

\[
\sigma_p = \alpha_1 Z^{n_1}
\]

(11)

\[
\varepsilon_p = \alpha_2 Z^{n_2}
\]

(12)
where, $\alpha_1, \alpha_2, n_1, n_2$ are constant, $\sigma_p$ and $\varepsilon_p$ are peak stress and strain. The dependence of peak stress and strain on Zener-Hollomon parameter is shown in figure 13. This diagram depicts that there is a linear relationship with the appropriate regression coefficient among the data.

Figure 11. Relation between peak stress versus peak strain rate at different temperatures (a) calculation of $n'$, (b) calculation of $\alpha$, (c) calculation of $n$ and (d) Arrhenius-type plot for activation energy for Al0.4 alloy. Data obtained from true stress-strain curves.
By calculating the parameters, the equations of peak stress and strain due to the Zener parameter are obtained as follows:

\[ \sigma_p = 1.79Z^{0.12} \]  
\[ \varepsilon_p = 3.48 \times 10^{-7}Z^{0.28} \]

Since the Zener parameter depends on the strain rate and deformation temperature, it can be observed that peak stress and peak strain are controlled by thermal processes [67]. As mentioned, the relationship between work hardening rate and true stress during the deformation process is of great importance with a physical meaning associated with structural change [40]. Density of the dislocations primarily investigates the work hardening rate parameter during plastic deformation. Therefore, dislocation assessment is very important in plastic deformation. What happens during the plastic deformation process for dislocation involves multiplicating the dislocation from straining and the annihilation of dislocation in recovery [42]. The linear relationship between the work hardening rate and the true stress related to the dislocation multiplication coefficient and the dislocation annihilation coefficient is as follows:

\[ \theta = \frac{\alpha GMb k}{2} - \frac{\Omega}{2} \sigma \]  
\[ \frac{\Omega}{2} \propto \frac{1}{Z} = 1 / \left[ \frac{\exp \left( \frac{Q_{\text{def}}}{RT} \right)}{Z} \right] \]

Therefore, by calculating the slope of the linear part of work hardening rate diagram due to true stress, the dislocation annihilation coefficient can be calculated. Information on the dislocation annihilation coefficient is given in table 4.

Table 3. Comparative values of activation energy of deformation for steels obtained by compressive testing.

| Steel            | Q(J mol\(^{-1}\)) | References | Steel            | Q(J mol\(^{-1}\)) | References |
|------------------|-------------------|------------|------------------|-------------------|------------|
| AISI W1          | 286               | [60]       | AISI A2          | 399               | [40]       |
| 25Mn8Al          | 300               | [61]       | H13              | 401               | [62]       |
| Al140            | 321               | [63]       | 25Mn6Al          | 405               | [61]       |
| EN9              | 332               | [63]       | 28CrMnTi         | 426               | [64]       |
| 17–4 PH          | 337               | [65]       | AISI D2          | 428               | [66]       |
| low carbon       | 340               | [59]       | AISI M2          | 455               | [40]       |
| VC9              | 355               | [63]       | MnCrCoFeNi       | 350               | [57]       |
| 25Mn              | 377               | [59]       | Al0.3CrCoFeNi   | 513               | [66]       |
| 25Mn3Al          | 397               | [59]       | Al0.4            | 434.1             | present work |

By calculating the parameters, the equations of peak stress and strain due to the Zener parameter are obtained as follows:

\[ \sigma_p = 1.79Z^{0.12} \]  
\[ \varepsilon_p = 3.48 \times 10^{-7}Z^{0.28} \]

Figure 12. The dependence of peak stress ln(sinh(\(\alpha\sigma_p\))) on Zener-Hollomon (lnZ), the data shows a linear dependence.
Figure 14 shows the relationship between the dislocation annihilation coefficient’s logarithm inversely with the temperature for Al0 and Al0.4 alloys. In this figure, the diagrams are divided into two linear parts: low temperature (blue line) and high temperature (red line). Since the slope of diagram represents the energy of deformation, the slope change of the line at 600 °C indicates the change of energy of deformation before and after this temperature. In other words, 600 °C is the temperature at which the deformation mechanism is likely to change. In fact, it is possible to study the deformation mechanism in the range of different temperatures by determining the deformation energy and examining the microstructure [68].

Another study in this regard is related to the calculation of steady-state stress. The following equation is one of the common and practical equations for this purpose. This equation determines the relationship between deformation temperature, strain rate, and steady-state stress [67].

\[
\sigma_{ss} = A_s \left( \dot{\varepsilon} \exp \left( \frac{Q}{RT} \right) \right)^q
\]

where \( \sigma_{ss} \) is the steady-state stress. Due to equation (17), it is clear that at a constant temperature, there is a linear relationship between the steady-state stress and the strain rate in the logarithmic scale, and if the strain rate is constant, a linear relationship between the logarithm of the steady-state stress and inversely with the temperature. Data on steady-state stress and temperature at a strain rate of 0.01/s is also given in table 4. At low temperatures, the stress at strain 0.4 was selected as the steady-state stress [68]. Due to the calculated activation energy of deformation, the values of q and A equal 0.1155 and 1.61, respectively. Therefore, the equation of steady-state stress at high temperature (T > 600 °C) is calculated as follows:

\[
\sigma_{ss} = 0.95 \left( \exp \left( \frac{52202.4}{T} \right) \right)^{0.12}
\]
Figure 15 shows the dependence of steady-state stress on temperature. The relationship between the data at low temperatures ($T \leq 600 \, ^\circ\text{C}$) and high temperatures ($T > 600 \, ^\circ\text{C}$) seems to be two lines with different slopes. Since slope of the line is related to the activation energy of deformation, it also changes by increasing the deformation temperature above 600 °C. It is clear that the activation energy of deformation is directly related to the temperature. Such behavior has also been reported in [68]. To calculate the activation energy of deformation at low temperatures, further tests at different strain rates must be performed.

Due to figures 14 and 15, it appears that 600 °C is a critical temperature for deformation. The study, which has been conducted from two perspectives of dislocation annihilation coefficients and steady-state stress, shows the difference of deformation energy at temperatures higher and lower than 600 °C. It seems that the deformation mechanism should also change depending on the temperature.

### 4. Conclusions

The present study focused on MnCrCoFeNi (Al0) and Al$_{0.4}$MnCrCoFeNi (Al0.4) alloys produced by vacuum melting and casting. High-temperature mechanical properties of these alloys were evaluated by hot

$$\sigma_{ss} = 1.61 Z^{0.12}$$  \hspace{1cm} (19)
compression test at different temperatures and strain rates. Results revealed that addition of Al to MnCrCoFeNi alloy reduced the diffusion rate and delayed recrystallization or increased its temperature, so that the initiating temperature of necklace structure increased by about 200 °C. It was also observed that stacking fault energy of Al0.4 alloy was also low, and the formation of nanoprecipitates (AlNi-rich B2) and nanotwins revealed that the dominant deformation mechanism was twin formation. Furthermore, significant changes in the yield strength of Al0.4 alloy with temperature were not observed, which was due to thermal stability of B2 precipitates. For Al0.4 alloy, the determined stress exponent and the activation energy of deformation were 6.86 and about 434 kJ mol⁻¹, respectively. In addition, the sluggish diffusion and the solution drag effects were related to B2 precipitates. The work hardening exponent for Al0.4 alloy increased, while it decreased for Al0 alloy for the temperatures up to 600 °C during the compression tests. In other words, the work hardening ability of Al0.4 alloy was revealed to be relatively higher than that of Al0 alloy. Moreover, studying deformation energy from both perspectives of dislocation annihilation coefficient and steady-state stress indicated that 600 °C was a critical temperature, above which the deformation activation energy changed.

Data availability statement
All data that support the findings of this study are included within the article (and any supplementary files).

ORCID iDs
Said Nategh  https://orcid.org/0000-0002-3002-317X
Reza Gholamipour  https://orcid.org/0000-0001-9109-5727

Figure 15. The effect of temperature on the steady state stress at 0.01/s strain rate for (a) Al0 and (b) Al0.4 alloy.
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