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Communication

Revealing the Role of Cross Slip for Serrated Plastic Deformation in Concentrated Solid Solutions at Cryogenic Temperatures

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Abstract: Serrated plastic deformation is an intense phenomenon in CoCrFeMnNi at and below 35 K with stress amplitudes in excess of 100 MPa. While previous publications have linked serrated deformation to dislocation pile ups at Lomer–Cottrell (LC) locks, there exist two alternate models on how the transition from continuous to serrated deformation occurs. One model correlates the transition to an exponential LC lock density–temperature variation. The second model attributes the transition to a decrease in cross-slip propensity based on temperature and dislocation density. In order to evaluate the validity of the models, a unique tensile deformation procedure with multiple temperature changes was carried out, analyzing stress amplitudes subsequent to temperature changes. The analysis provides evidence that the apparent density of LC locks does not massively change with temperature. Instead, the serrated plastic deformation is likely related to cross-slip propensity.

Keywords: cryogenic deformation; serrations; high-entropy alloys

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1. Introduction

Metallic materials have shown serrated plastic deformation at different temperatures [1–4]. Recent publications highlight especially intense serrated deformation in high-entropy alloys (HEA) [5–8]. Equiatomic CoCrFeMnNi even exhibits low temperature serrations at 35 K, a temperature higher than previously reported for any other metal or alloy [6]. While a few different hypotheses exist to explain low temperature serrations [9–11], recent results led to a phenomenological model based on dislocation pile ups at LC locks [5,6]. Alternate hypotheses, related to a thermomechanical [9], twinning or martensite-based instability [11] were previously invalidated and are neglected in this communication [5–7].

The model presented in Ref. [6] extends the work of Seeger [10], based on screw dislocation immobility in close-packed crystals at low temperatures [12]. As mobile screw dislocations interact with forest dislocations, they form immobile jogs [12,13]. This restriction is not seen for edge dislocations, for a more detailed view on the differences between edge and screw interaction with forest dislocations refer to Figure 6 in Ref. [6]. The motion of these jogged screw dislocations is associated with vacancy formation [12,13] which becomes increasingly difficult with decreasing temperature. Thus, at temperatures close to 0 K, the motion of jogged screw dislocations is restricted so much that it compromises cross-slip propensity. Correspondingly, as opposed to dislocations cross-slipping out of pile ups at barriers such as LC locks, they would activate dislocation sources on the other side of the pile up as a result of the stress-field at its head [10]. This proliferation and motion of dislocations leads to a macroscopic stress drop, seen as a serration. Ref. [6]

extends the model, accounting for cross-slip propensity as a function of temperature and dislocation density, along with an explanation for transition from continuous to serrated plastic deformation as temperature decreases. In contrast, the original Seeger model [10,14] considers only a critical temperature condition associated with serrations, establishing that at a given temperature, deformation would either be continuous or serrated in nature. It cannot explain initially continuous and subsequently discontinuous deformation, as seen in CoCrFeMnNi for example [6]. This is circumvented by either considering (i) the aforementioned cross-slip propensity variation [6] or, alternatively, (ii) assuming an exponential LC lock density–temperature variation as proposed in Ref. [12]. The present work investigates the validity of these two possibilities by the application of a specific mechanical testing scheme at low temperatures [6,15]. The in situ observation of cross-slip events or the LC lock density as fundamental requisites of the two alternatives exhibits multiple complications when performed at such low temperatures, while microstructural investigations post deformation suffer from partial recovery of dislocation features on heat-up. These issues are presently avoided with a novel interrupted tensile test where serration characteristics are evaluated through stress drop amplitude ($\Delta\sigma_e$). The details of this test are as described in the following section and have been designed specifically for this investigation. Correspondingly, issues that cannot be avoided in other methods are avoided.

2. Materials and Methods

CoCrFeMnNi was synthesized by arc melting of high-purity elements. It was cast, homogenized at 1200 °C for 72 h, rotary swaged, machined and annealed at 800 °C for 1 h. For a detailed overview, please refer to Ref. [16].

Interrupted tensile deformation was carried out between 25 K and 8 K in a sealed chamber with He vapor at ~50 mbar, with multiple interruptions (sequence of events and associated temperatures and strains stated in later sections). The machine used for this was the MTS25 (MTS Systems, Eden Prairie, USA) with a maximum load of 25 kN [17]. The extension was measured by a pair of clip-on extensometers. The specimens had a cylindrical gauge section of 22 mm in length and 4 mm in diameter. Tensile testing was performed at a constant crosshead speed equaling an initial plastic strain rate of ~3×10^{-4} s^{-1}. The strain was measured using two strain gauges within the gauge length of the specimens. Data analysis was carried out using force, time and elongation results through the proprietary software packages Origin 2020b by OriginLab and MATLAB R2018a (MathWorks). For more information, please refer to Ref. [6].

3. Results and Discussion

3.1. Tensile Tests up to Fracture Model Considerations

Figure 1a shows serrated plastic deformation of CoCrFeMnNi at 8, 15 and 25 K, as seen in the engineering stress–strain ($\sigma_e - \varepsilon_e$) curves for tensile tests. Corresponding $\Delta\sigma_e$ were determined from the difference between stress maxima and minima for each serration (Figure 1b). As deformation continues, the intensity of serrations as measured by $\Delta\sigma_e$ increases. Additionally, serrated plastic deformation is initiated at a lower strain at lower temperatures.

According to the model of serrated plastic deformation described above, dislocations pile up at LC locks; at low temperatures, dislocation sources are activated and massive dislocation proliferation events take place at the heads of the pile ups, seen as macroscopic stress drops [10]. This is only noted at low temperatures since mobility of intersected screw dislocations and cross slip is restricted for close-packed crystals at very low temperatures [10,12]. However, this model proved insufficient in explaining results where deformation was continuous at lower strains and discontinuous at larger strains, since the condition was based only on temperature. Different alloys have shown a transition from continuous, to partially serrated and finally fully serrated deformation as temperature decreases in the range T < 50 K [6,14]. Skoczeń et al. [15] proposed that the LC lock density increases exponentially with decreasing temperature to satisfy these results. Correspondingly, the number of pile ups and dislocation proliferation events would increase with decreasing temperature. Alternatively, multiple factors affecting cross slip were instead considered to explain these results in Ref. [6]. These two more recent versions of the model are explained below:

(i) Skoczeń et al. [15] have proposed a rapid increase in LC lock formation rate $\widetilde{F}^{+}_{LC}$ during deformation with decreasing temperature and assigned it the form:

$$\lg\widetilde{F}^{+}_{LC} = A + B\lg T \quad (1)$$

Here, A and B are constants and T is the temperature (note: B is negative due to the inverse variation of $\widetilde{F}^{+}_{LC}$ and T). $\widetilde{F}^{+}_{LC}$ expectedly changes by orders of magnitude below 40 K [15]. A minimum strain ε_{min} is required to generate a sufficient LC lock density and dislocation pile ups which result in serrated deformation. At higher temperatures (25 K), $\widetilde{F}^{+}_{LC}$ is lower, thus, ε_{min} is necessarily larger. At lower temperatures (8 K), the ε_{min} instead decreases significantly due to much higher $\widetilde{F}^{+}_{LC}$.

(ii) Ref. [6] considers screw dislocation mobility [10], stacking-fault energy (SFE) and dislocation pile up characteristics and their effect on cross-slip propensity. Dislocation density and temperature affect this most significantly. At higher temperatures (i.e., 25 K presently), cross slip is facilitated easily and proceeds sufficiently until a critical strain (ε_{min}), where dislocation density is high enough to compromise screw dislocation mobility, subsequently resulting in serrations. At lower temperatures (8 K), cross slip is so severely restricted that even minor strain results in serrations. Notably, this model considers LC lock density to scale with dislocation density and assumes it to be similar in the temperature range of 8–25 K.

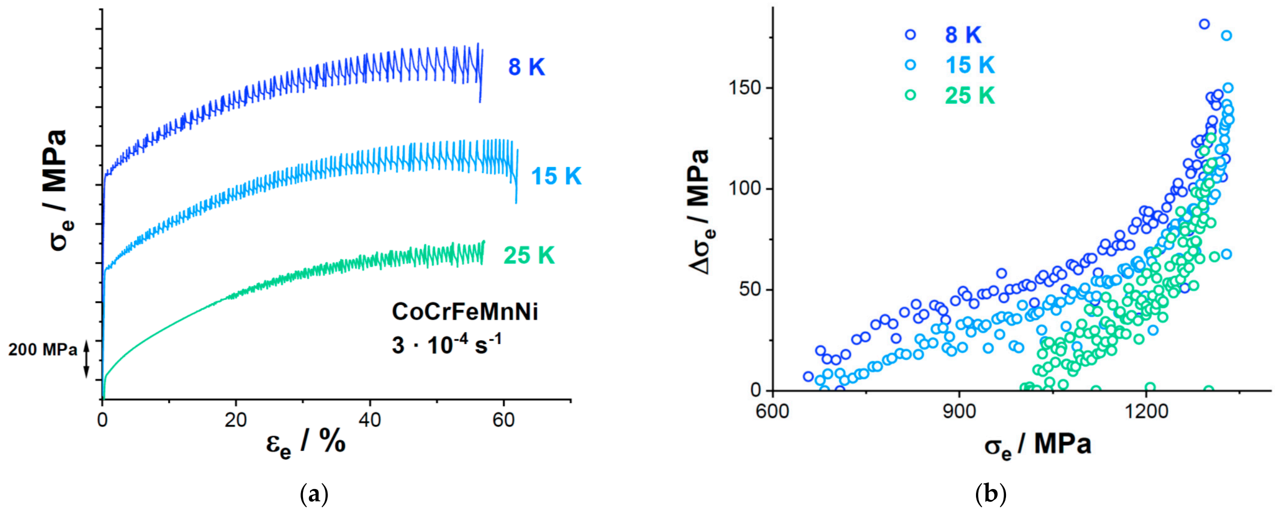


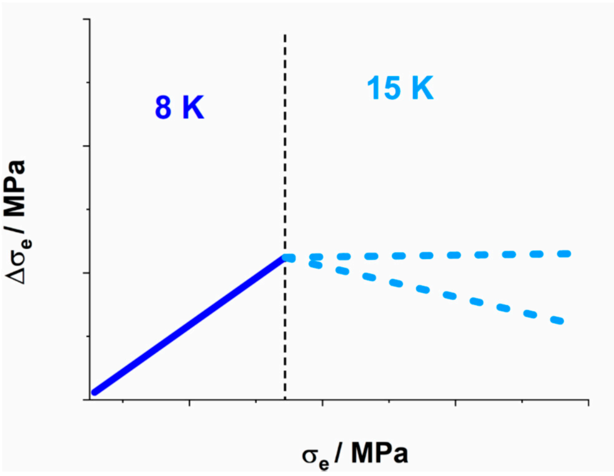
Figure 1. (**a**) $\sigma_e - \varepsilon_e$ plots of CoCrFeMnNi at 8, 15 and 25 K. (**b**) $\Delta\sigma_e - \sigma_e$ plots of serrations. Data from [6]. The data in (**a**) is offset along the vertical axis so that each curve can be clearly distinguished.

3.2. Expectations on Interrupted Tests from the Different Models

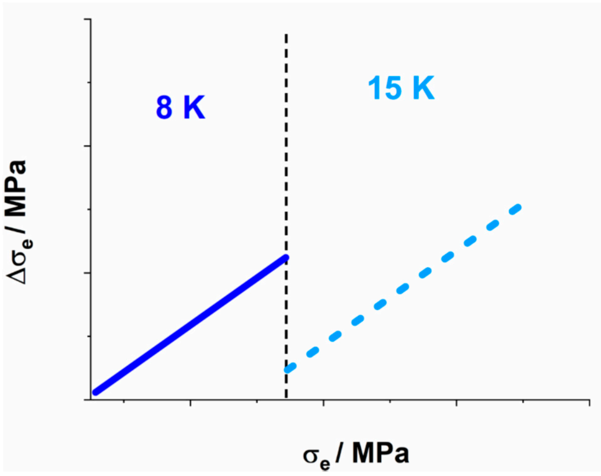
To test these models, a temperature change test is carried out. The tensile test is initiated at 8 K and the sample is strained to an engineering strain of approximately $\varepsilon_e \sim 10\%$ and then unloaded. The temperature is then changed to 15 K and the test continued. Stress drop amplitude vs. stress $\Delta\sigma_e - \sigma_e$ is evaluated to confirm the correct model. Regardless of the model, the initial trend at 8 K should have a positive variation, as seen in the experimental data from the uninterrupted test in Figure 1b. Post-interruption, there are two possibilities as illustrated in Figure 2.

(i) Figure 2a shows a condition where instability is met by some physical factor, e.g., minimum dislocation or LC lock density. At 8 K, the necessary LC lock density for serrated plastic deformation is achieved easily. On changing the temperature to 15 K, the LC lock density remains unaffected, hence serrated deformation proceeds onwards. However, based on the lower $\widetilde{F}^{+}_{LC}$, the $\Delta\sigma_e - \sigma_e$ slope may be lower than at 8 K, possibly even plateauing. This is the generalized interpretation of Ref. [15].

(ii) Alternatively, instability is controlled by cross-slip propensity and, correspondingly, dislocation density and temperature. Thus, after the interruption, $\Delta\sigma_e$ should drop significantly, since cross-slip probability is greater at 15 K than 8 K, evidenced by $\Delta\sigma_e - \sigma_e$ variation (Figure 1b). Since dislocation density increases during further deformation, $\Delta\sigma_e$ should subsequently increase consistently with σ_e (seen in Figure 2b). Thus, the important difference between the expected results is that in one model the $\Delta\sigma_e$ values pre- and post-interruption match [15] and in the other model, there is a distinct change in $\Delta\sigma_e$ [6].



(a)



(b)

Figure 2. Schematic $\Delta\sigma_e - \sigma_e$ trend for tensile tests with an interrupted temperature change. Both (**a**) and (**b**) show possible interpretations of the temperature change based on the models adapted from [6,15], respectively.

3.3. Results of the Interrupted Tests

The temperature change tensile test consists of interruptions at $\varepsilon_e \sim 10\%$, 22% and 40%. The test begins at 8 K and then continues at 15, 25 and back at 8 K after the respective interruptions to verify consistent trends despite pre-deformation. $\sigma_e - \varepsilon_e$ and corresponding $\Delta\sigma_e - \sigma_e$ results are shown in Figure 3.

Figure 3b is clearly indicative of the trend expected for the cross-slip propensity model (compare with Figure 2b). A $\Delta\sigma_e$ drop is noted for temperature changes to both 15 and 25 K. As a final verification, reloading at 8 K would result in a drastically higher $\Delta\sigma_e$ post-interruption when considering cross slip [6] in comparison to a lower $\Delta\sigma_e$ when considering $\widetilde{F}^{+}_{LC}$ from Equation (1) [15]. The severe increase in $\Delta\sigma_e$ provides further evidence of a cross-slip-based mechanism.

The $\Delta\sigma_e - \sigma_e$ variation of the uninterrupted tests has been included in Figure 3b. The $\Delta\sigma_e - \sigma_e$ values are noticeably lower after the interruptions and temperature changes to 15 and 25 K. This may be explained by the stress τ_{bow} to move the unpinned portion of a jogged screw dislocation using G (shear modulus), b (Burgers vector), l_0 (mobile dislocation length between the jogs) and α (constant ~0.1–0.2) [12]:

$$\tau_{bow} = \frac{\alpha\, G\, b}{l_0} \quad (2)$$

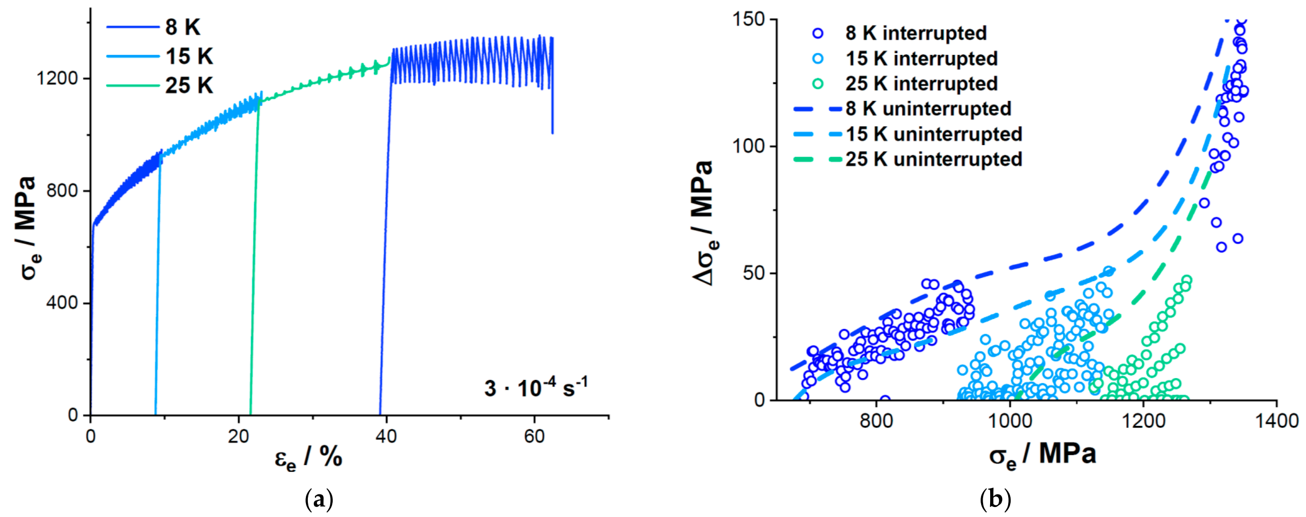


Figure 3. (**a**) $\sigma_e - \varepsilon_e$ for tensile test at multiple temperatures and (**b**) the corresponding $\Delta\sigma_e - \sigma_e$ plot. The dashed lines in (**b**) represent the curves for the uninterrupted test in Figure 1.

As the temperature decreases, the minimum l_0 for a mobile screw dislocation keeps increasing. At a given temperature, screw dislocations of a length less than some l_0 are immobile and at higher temperatures the minimum l_0 is shorter. Accordingly, several dislocations are likely immobile at 8 K but at 15 K the critical l_0 being shorter, the same dislocations become mobile. Combined with the higher stress, cross slip is significantly more viable subsequent to the interruption and reloading at a higher temperature, making serrations less intense. Additionally, since there is an unloading step, unstressed dislocations only partially recede from the stressed state due to dislocation–dislocation interactions. Thus, the uninterrupted and interrupted $\Delta\sigma_e - \sigma_e$ trends should be similar but offset by different states of dislocations.

In the given test, the observed drop in $\Delta\sigma_e$ can only be explained in the absence of an exponential variation of dislocation or LC lock density with temperature. Hence, this experiment reinforces the model given in Ref. [6] where cross slip based on temperature, dislocation density and dislocation mobility govern the serration behavior close to 0 K in face centered cubic alloys.

4. Conclusions

The $\Delta\sigma_e - \sigma_e$ trends observed in tensile tests with deliberate, intermediate temperature steps conducted on CoCrFeMnNi experimentally verify that low temperature serrations in face centered cubic high-entropy alloys are governed by the immobility of screw dislocations. The temperature-dependent cross-slip propensity and dislocation density throughout deformation are the relevant parameters controlling the immobility.

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