

HYDROGEN-INDUCED DEFORMATION MECHANISM TRANSITION REVEALED BY ACOUSTIC EMISSION DURING TENSILE DEFORMATION OF AN FCC HEA

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Abstract

Hydrogen can significantly influence the plastic deformation behaviour of high-entropy alloys (HEAs) by altering the dominant deformation mechanisms. In this work, the tensile deformation of a face-centred cubic (FCC) $\text{Al}_{0.35}\text{CoCrFeNi}$ alloy was investigated using acoustic emission (AE) analysis before and after hydrogen charging, with a focus on statistical evaluation of deformation mechanisms. The mechanical response shows only minor changes after hydrogen charging, with a slight increase in yield strength ($R_{p0.2}$ from 214 ± 2 MPa to 228 ± 3 MPa) and ultimate tensile strength (R_m from 576 ± 3 MPa to 587 ± 5 MPa), while ductility remains nearly unchanged (elongation $\sim 65\%$). Despite these small differences, the AE response reveals a pronounced transition in deformation behaviour. Hydrogen charging under tensile loading leads to a significant increase in AE activity and promotes a transition from twinning-dominated to dislocation-controlled plasticity. This transition is accompanied by a higher occurrence of high-energy AE events, indicating more intense and localized deformation processes. Statistical analysis confirms that these changes are highly significant and that hydrogen markedly increases the likelihood of dislocation-related AE events. The observed behaviour is consistent with hydrogen enhanced localized plasticity (HELP), suggesting that hydrogen under tensile loading enhances localized dislocation movement while reducing the contribution of competing mechanisms such as twinning. These results demonstrate that acoustic emission combined with statistical analysis provides a sensitive and reliable tool for identifying hydrogen-induced changes in deformation mechanisms, even when conventional mechanical properties remain largely unaffected.

Keywords: High entropy alloy, acoustic emission, tensile deformation, statistical analysis, hydrogen enhanced localized plasticity

1. INTRODUCTION

Face-centred cubic (FCC) high-entropy alloys (HEAs), particularly those based on the Co–Cr–Fe–Ni system, exhibit excellent mechanical properties and complex deformation behaviour governed by the interplay between dislocation slip and deformation twinning [1-6]. The balance between these mechanisms is sensitive to external factors, including hydrogen, which may significantly alter the dominant deformation mode [7-10]. One of the most widely accepted mechanisms explaining this behaviour is hydrogen enhanced localized plasticity (HELP), which attributes hydrogen-induced effects to enhanced dislocation mobility and localized plastic flow [7].

Acoustic emission (AE) is a highly sensitive technique for real-time monitoring of deformation processes, capable of distinguishing between different deformation mechanisms based on signal characteristics [11-16]. However, most studies rely on a combination of AE and microstructural analysis, while the direct statistical evaluation of AE signals as a standalone tool remains limited. This limitation becomes critical in cases where hydrogen does not significantly change conventional mechanical properties, making its effect difficult to detect using standard mechanical testing. In such cases, AE provides a more sensitive insight into underlying deformation mechanisms.

The present work investigates tensile deformation of an FCC HEA before and after hydrogen charging, with emphasis on the statistical analysis of AE signals associated with dislocation movement and twinning. Although hydrogen charging leads to only minor changes in mechanical properties, it fundamentally alters the dominant deformation mechanisms, as revealed by AE analysis. The novelty of this study lies in the application of statistical methods, including significance testing and effect size analysis, to AE data, demonstrating that AE can reliably identify hydrogen-induced transitions between deformation mechanisms.

2. EXPERIMENTAL PROCEDURE

2.1 Casting, heat treatment and hot forging

The Al_{0.35}CoCrFeNi alloy was prepared by vacuum induction melting of pure elements (Co, Cr, Fe, Ni, Al) in a ceramic crucible, followed by casting into a graphite mould under argon atmosphere. The melt was held at 1600 °C for 10 min to ensure chemical homogeneity. The resulting ingots (ϕ 23.8 mm, length 150 mm) were sectioned into smaller specimens.

To eliminate dendritic segregation, the cast alloy was solution annealed at 1340 °C for 6 h under argon, followed by cooling to room temperature. Grain refinement was achieved by three-step hot forging (30-60-80% of machine energy) using a screw press FICEP PVX 160 (max. 17 000 J), with intermediate annealing at 1250 °C for 20 min between deformation steps. Final heat treatment was carried out at 1320 °C for 30 min followed by air cooling.

2.2 Microstructure characterization and hydrogen charging

The microstructure was characterized using light microscopy (LM) and laser scanning confocal microscopy (LSM). Standard metallographic preparation, including grinding, polishing, and electropolishing, was applied. Grain size was evaluated using image analysis software and statistically processed.

Hydrogen charging of heat-treated samples was performed by cathodic charging at room temperature for 24 h in an H₂SO₄-based electrolyte with a current density of 0.6 mA/mm². The hydrogen content was subsequently measured using thermal desorption spectrometry (TDS) by a Bruker G4 Phoenix analyser.

2.3 Mechanical testing

Tensile specimens (gauge diameter 4.5 mm, length 26 mm) were machined from heat-treated material and tested at room temperature according to ISO 6892-1:2019 at a strain rate of $2.5 \times 10^{-4} \text{ s}^{-1}$ using a Zwick/Roell Z100 machine.

AE signals were recorded during tensile deformation using a piezoelectric sensor (400–800 kHz). The sensor was attached using vacuum grease, and signals were pre-amplified by 40 dB. Data acquisition was performed using an AMSY-5 system at a sampling rate of 20 MHz, with a threshold of 40 dB.

3. RESULTS AND DISCUSSION

Figure 1(a) shows the hot-forged condition, while **Figure 1(b)** shows the heat-treated state of Al_{0.35}CoCrFeNi alloy exhibited a homogeneous single-phase FCC microstructure with equiaxed grains and occasional annealing twins [11]. The average grain size and shape factor after heat treatment were measured to be 500 µm and 0.63, respectively. Cathodic hydrogen charging for 24 hours resulted in a hydrogen content of 10.79 ppm measured by TDS. No visible surface damage or cracking was observed after charging, allowing immediate mechanical testing.

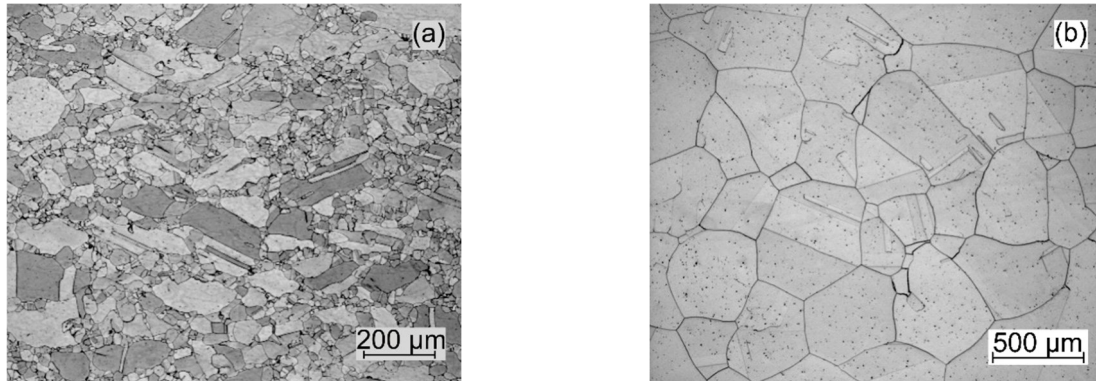


Figure 1 Microstructure of studied $\text{Al}_{0.35}\text{CoCrFeNi}$ HEA: (a) hot-forged (LSM) and (b) heat-treated state (LM)

The evolution of AE activity during tensile deformation of the FCC $\text{Al}_{0.35}\text{CoCrFeNi}$ alloy is shown in **Figure 2** for both hydrogen-uncharged and hydrogen-charged specimens. The mechanical response of both conditions is very similar. The hydrogen-uncharged specimen exhibits a yield strength $R_{p0.2} = 214 \pm 2$ MPa, ultimate tensile strength $R_m = 576 \pm 3$ MPa, and elongation of 65.9%. After hydrogen charging, a slight increase in strength is observed, with $R_{p0.2} = 228 \pm 3$ MPa and $R_m = 587 \pm 5$ MPa, while the elongation remains nearly unchanged (64.9%). These results indicate that hydrogen has only a minor effect on the conventional mechanical properties of the investigated FCC HEA.

Despite the similar mechanical behaviour, the acoustic emission response differs significantly between the two conditions. The hydrogen-charged specimen exhibits a substantially higher number of AE events and increased signal activity throughout plastic deformation compared to the uncharged condition.

These results indicate that hydrogen modifies the underlying deformation mechanisms despite minimal changes in macroscopic properties. The enhanced AE activity in the hydrogen-charged state indicates more intensive and dynamic plastic deformation, which is consistent with increased dislocation mobility.

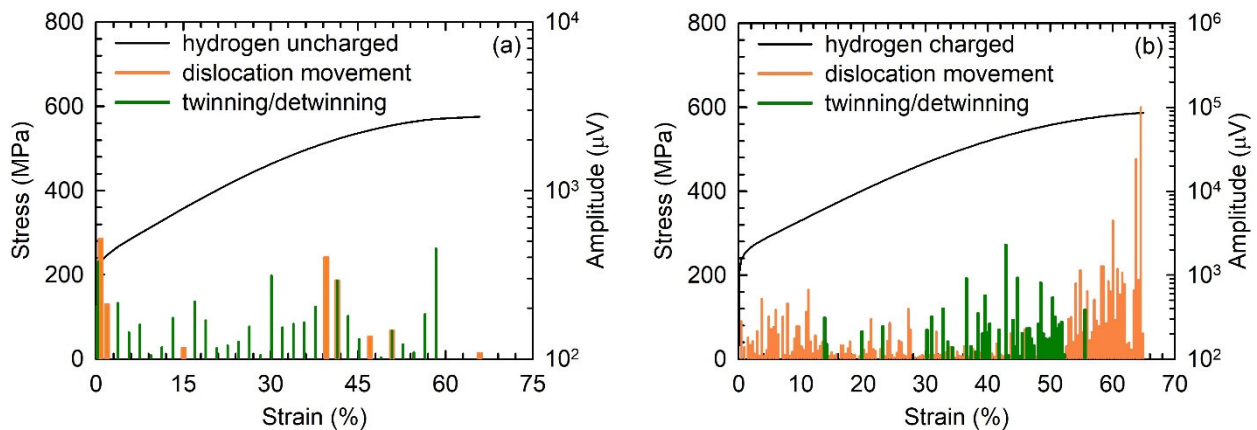


Figure 2 The evolution of acoustic signals together with the stress-strain curves of hydrogen (a) uncharged and (b) charged specimens

Figure 3 shows the scaling relationship between energy (E) and amplitude (A) of AE signals for hydrogen-uncharged and hydrogen-charged specimens. The data follow a power-law relation of the form $E_i = S_i A_i^2$, where the parameter S reflects the position of individual branches in the log-log representation [11–16]. Two distinct parallel branches can be identified in both conditions, indicating the presence of two dominant types of AE sources. To quantify this behaviour, the slope and intercept of the $\log(E)$ – $\log(A)$ relationship were determined, and the data points were projected onto the fitted line. The distribution of the projected data was

fitted using a double Gaussian function, where each Gaussian corresponds to one branch of AE signals, as illustrated in the insets of **Figures 3(a)** and **(b)**. The intersection of the two distributions was used to separate the branches, while the positions of their maxima define the characteristic values of the parameter S . These distributions further allow estimation of the mean values and standard deviations for each branch. The parameter S was used solely for branch identification and was not further considered in the subsequent analysis.

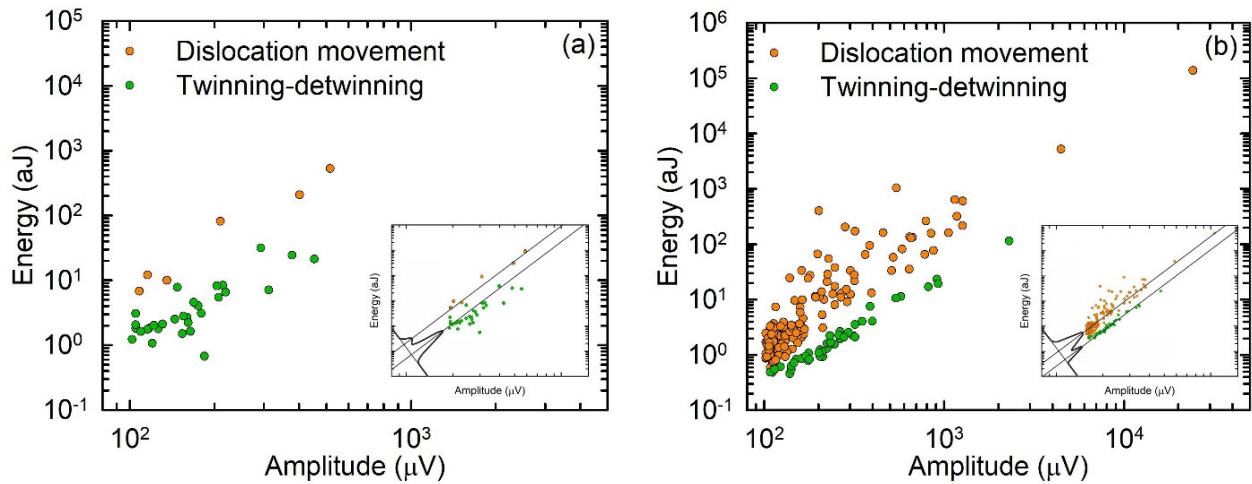


Figure 3 Two parallel branches of energy-amplitude scaling of AE signals for hydrogen (a) uncharged [11] and (b) charged specimens. The insets in figures show the distribution of the projected data points follows a double Gaussian distribution, in which each Gaussian distribution represents one branch of the AE signals

Figure 4 presents the maximum likelihood (ML) analysis of AE signals for hydrogen-uncharged and hydrogen-charged specimens. Two parallel branches are identified in both conditions (orange and green), consistent with the energy–amplitude scaling shown in **Figure 3**. The ML analysis confirms that the AE signals follow avalanche behaviour characterized by a power-law distribution $P(E) \sim E^{-\varepsilon}$ [11]. For the hydrogen-uncharged specimen, the energy exponents are $\varepsilon_{disl} = 1.53 \pm 0.07$ and $\varepsilon_{twin} = 2.07 \pm 0.24$ [11], while for the hydrogen-charged condition they decrease to $\varepsilon_{disl} = 1.43 \pm 0.04$ and $\varepsilon_{twin} = 1.83 \pm 0.13$.

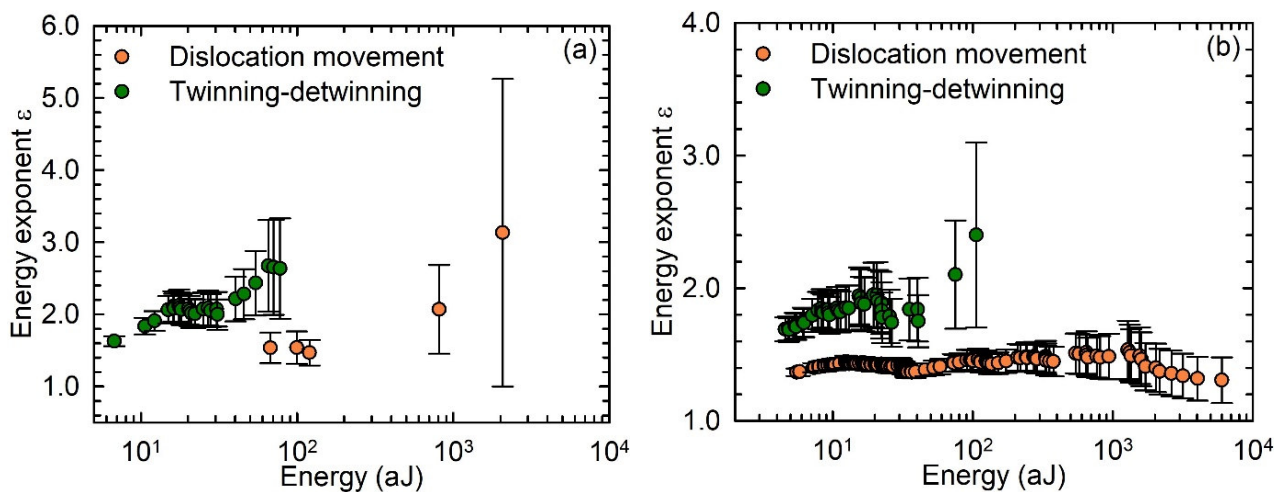


Figure 4 ML analysis of hydrogen (a) uncharged [11] and (b) charged specimens

Lower exponent values for dislocation-related signals indicate an increased probability of high-energy events. This is supported by the observed energy ranges, where dislocation-related AE signals span from approximately 10^1 to 10^4 aJ in the uncharged state and extend from 10^0 to 10^4 aJ after hydrogen charging. In contrast, twinning-related signals remain confined to a lower energy range of approximately 10^0 to 10^2 aJ for both conditions. The shift towards lower exponent values in the hydrogen-charged state indicates an increased occurrence of high-energy deformation bursts. This behaviour is consistent with HELP, where hydrogen promotes localized dislocation movement and facilitates collective dislocation activity.

The statistical analysis of AE event (**Table 1**) distributions reveals a pronounced effect of hydrogen on the dominant deformation mechanisms. In the hydrogen-uncharged condition, twinning-related events dominate ($\approx 78.9\%$), while dislocation-related signals represent only $\approx 21.1\%$. After hydrogen charging, this ratio is reversed, with dislocation-related events increasing to $\approx 76.0\%$ and twinning reduced to $\approx 24.0\%$.

Statistical analysis (chi-square test, Fisher's exact test, Cramér's V and odds ratio) was performed using standard methods described in [17–20]. The transition is highly significant, as confirmed by the chi-square test ($\chi^2 = 42.16$, $p < 0.0001$) and Fisher's exact test ($p < 0.0001$). The strength of the association is supported by Cramér's V ($V = 0.445$), indicating a strong relationship between hydrogen presence and the active deformation mechanism. Furthermore, the odds ratio (OR ≈ 11.9 , 95% CI: 4.9–28.8) shows that hydrogen increases the probability of dislocation-related AE events by more than an order of magnitude.

These results demonstrate that hydrogen induces a statistically significant transition from twinning-dominated to dislocation-dominated plastic deformation, providing strong evidence for HELP.

Table 1 AE event distribution

Sample	Dislocations	Twinning/detwinning	Row total	Twin fraction
uncharged	8	30	38	78.9%
charged	133	42	175	24.0%
Column total	141	72	213	

4. CONCLUSION

A hydrogen-induced transition in deformation mechanisms during tensile deformation of an FCC Al_{0.35}CoCrFeNi alloy was identified using acoustic emission. The following conclusions can be added:

1. Hydrogen charging has only a minor effect on mechanical properties, with slightly increased strength and nearly unchanged ductility.
2. A pronounced reduction of twinning activity is observed, decreasing from $\approx 78.9\%$ in the uncharged condition to $\approx 24.0\%$ after hydrogen charging. This change is accompanied by a significant increase in the total number of AE events and a shift toward dislocation-controlled plasticity of the hydrogen-charged specimen during tensile deformation.
3. Statistical analysis ($\chi^2 = 42.16$, $p < 0.0001$; OR ≈ 11.9) confirms that hydrogen significantly alters the active deformation mechanisms, consistent with HELP.

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