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Heterostructure High Entropy Alloys in the CoNiFeMn–Mo System Intended for Deep Drawing Applications

Kamil Cichocki[1,a*], Anna Wójcik[1,b], Paulina Lisiecka-Graca[1,c], Tymon Warski[2,d] and Krzysztof Muszka[1,e]

[1]AGH University of Kraków, Department of Metal Forming and Metallurgical Engineering, Kraków, Poland

[2]Łukasiewicz Research Network - Institute of Non-Ferrous Metals, 5 Sowińskiego Street, 44-100 Gliwice, Poland

[a*]cichocki@agh.edu.pl, [b]anwojcik@agh.edu.pl, [c]graca@agh.edu.pl, [d]tymon.warski@gmail.com, [e]muszka@agh.edu.pl

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Abstract. High-entropy alloys (HEAs), owing to their exceptionally favourable strength–ductility balance, are regarded as promising candidates for applications in the energy, automotive, and aerospace industries. A defining characteristic of face-centered cubic (FCC) high-entropy alloys is their low stacking fault energy, which facilitates deformation via mechanical twinning and promotes the activation of transformation-induced plasticity (TRIP) and twinning-induced plasticity (TWIP) mechanisms. The present study focuses on the development of a heterostructured material composed of CoNiFeMn and $(CoNiFeMn)_{95}Mo_5$ alloys. Furthermore, the Erichsen cupping test was performed to assess the formability of the produced material and to evaluate its suitability for deep drawing applications.

Introduction

High-entropy alloys (HEAs) have attracted significant research interest in recent years. Their concept, based on pronounced chemical disorder, renders these materials competitive with conventional alloys that are typically based on a single principal element[1], [2]. Despite their complex chemical compositions, HEAs tend to form stable solid solutions owing to the high configurational entropy of mixing, which reduces the Gibbs free energy of the solid solution. As a result, the formation of intermetallic phases is often suppressed[3]. However, HEAs with a face-centered cubic (FCC) crystal structure are generally characterized by a relatively low yield strength combined with excellent ductility. Conventional approaches for increasing yield strength, such as grain refinement[4] or precipitation strengthening[5], usually lead to a pronounced reduction in ductility[6]. Another important characteristic of these alloys is their low stacking fault energy (SFE)[7], [8] which promotes deformation by twinning and consequently leads to the activation of the twinning induced plasticity effect (TWIP)[9], [10], [11].

One approach to improving the mechanical properties of high-entropy alloys is the development of heterostructured materials. Several methods exist to achieve heterostructured architectures, including heterogeneous gradient structure [12], heterogeneous grain structure[4] and heterogeneous phase structure[13].

The main aim of the present study was to fabricate a heterostructured material via cold rolling followed by static recrystallization, thereby combining heterogeneous grain structures with the precipitation of the intermetallic μ phase. In addition, the study seeks to evaluate the formability of the produced materials under deep-drawing conditions. For this purpose, two materials were selected: CoNiFeMn and a molybdenum-alloyed variant, $(CoNiFeMn)_{95}Mo_5$, both exhibiting a propensity for deformation by twinning, with the addition of molybdenum further enhancing twinning activity [14]. Our previous studies have demonstrated that during recrystallization annealing of $(CoNiFeMn)_{95}Mo_5$, precipitation of the μ phase occurs; in the present work, this phase was utilized as a strengthening constituent[15] and also investigate the influence of intermetallic phase for deep-drawing process.

Experimental Procedure

In the present study, the feasibility of producing materials with a heterogeneous microstructure in CoNiFeMn and $(CoNiFeMn)_{95}Mo_5$ high entropy alloys (HEAs) was investigated. The alloys were synthesized by melting in an open induction furnace under a continuous flow of inert gas (argon) directed onto the surface of the molten metal. The total weight of each ingot was 10 kg. The ingots were produced from constituent elements with a purity of 99.95%. The chemical compositions of the as-cast alloys are listed in Table 1.

Subsequently, the alloys were subjected to a homogenization heat treatment at 1200 °C for 24 h. For further characterization, the materials underwent thermomechanical processing according to the schedule shown in Fig. 1. The chemical composition was analyzed using scanning electron microscopy equipped with energy-dispersive X-ray spectroscopy (SEM-EDS). Microstructural characterization, as well as texture analysis after selected stages of deformation and annealing, was performed using scanning electron microscopy combined with electron backscatter diffraction (SEM–EBSD) with post-processing using software OIM TSL 8. All microstructural and texture analyses were conducted using a NanoNova SEM 450 scanning electron microscope.

Table 1. Chemical compositions of the investigated materials in at%.

Alloy	Abbrev.	Fe	Mn	Ni	Co	Mo
CoNiFeMn	Mo0	24.7	24.2	25.5	25.6	0
$(CoNiFeMn)_{95}Mo_5$	Mo5	23.6	22.5	24.2	24.5	5.2

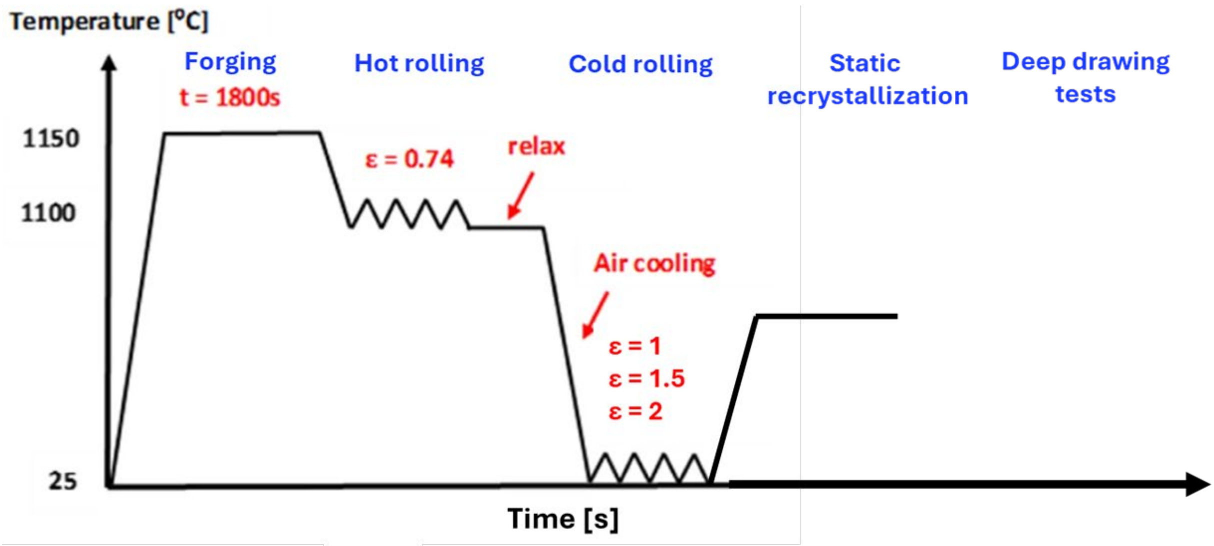

Fig. 1. Scheme of thermomechanical processing of studied alloys.

Microstructural analyses were performed on materials after hot rolling, which were considered to represent the initial condition, and subsequently after cold rolling to true strains of $\varepsilon = 1.0$, 1.5, and 2.0. In the present work, the feasibility of producing a heterostructured material through cold rolling followed by static recrystallization was investigated. The cold-rolled materials were subsequently subjected to recrystallization annealing.

The present study was focused on the possibilities to use heterostructured HEA materials deep drawing process. For this purpose, the Erichsen IE_{21} cupping test (in accordance with EN ISO 20482) was performed. Square sheet specimens with dimensions of 65 × 65 mm and a nominal thickness of 1.5 mm were prepared from the selected material variants. The test was carried out at a constant punch velocity of 10 mm/s using a hemispherical punch with a diameter of 14.98 mm. Tests were conducted three times for each variant.

To determine the mechanical properties of the materials selected for the forming tests, uniaxial tensile tests were conducted. Dog-bone shaped specimens were prepared, and the test was carried out using a Zwick 250 testing machine. Two specimens were tested for each variant.

Results and Discussion

Fig. 2 presents the microstructures of the CoNiFeMn and $(CoNiFeMn)_{95}Mo_5$ alloys after hot rolling. The microstructures of both materials are characterized by equiaxed grains with a high density of annealing twins. The difference in grain size observed between the two alloys arises from the effect of molybdenum addition on the kinetics of dynamic recrystallization as well as grain growth, resulting in a retardation of these processes. This phenomenon has been discussed in greater details in previous studies [15], [16].

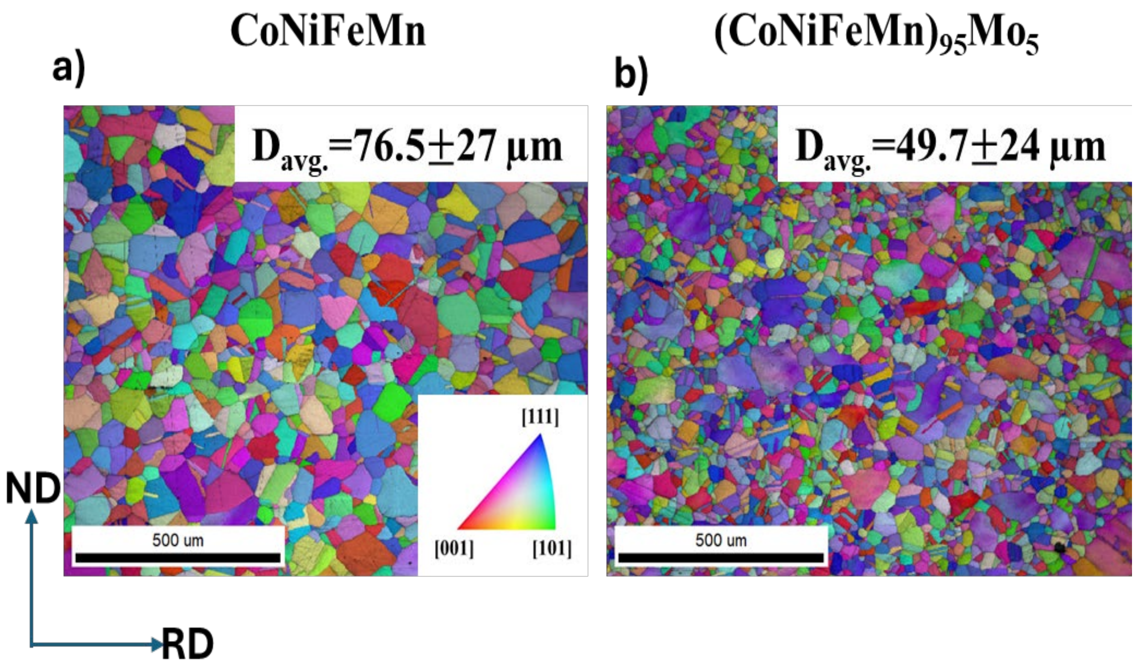

Fig. 2. Inverse pole figure maps for a) CoNiFeMn and b) $(CoNiFeMn)_{95}Mo_5$ alloys.

The initial material exhibiting the microstructure shown in Fig. 2 was subsequently subjected to cold rolling to accumulated true strains of ε = 1.0, 1.5, and 2.0. The microstructures of the materials at different deformation levels are presented in Fig. 3. All materials exhibit a microstructure typical for cold rolling, characterized by strongly elongated grains along the rolling direction. Deformation twins are also observed in the microstructure. In the case of the CoNiFeMn alloy, deformation twins become clearly visible at a strain of ε = 1.5, whereas in the $(CoNiFeMn)_{95}Mo_5$ alloy they can be observed at all considered strain levels.

Pole figure analysis (Fig. 3) confirms the development of a strong crystallographic texture with increasing deformation, as evidenced by the shift of intensity maxima corresponding to individual texture components. For both alloys, the evolution of texture is qualitatively similar; however, differences between the alloys are reflected primarily in the intensity of the developed texture. At a strain of ε = 1.0, the dominant texture components in both materials are the Brass {011}⟨211⟩ and rotated Goss {110}⟨110⟩ components, which are indicative of deformation dominated by twinning mechanisms. With increasing strain, the intensity of the Brass component decreases, while the rotated Goss component becomes more pronounced. Additionally, the emergence of A {110}⟨111⟩ and P {011}⟨211⟩ texture components is observed, further confirming the activation of deformation twinning. This behaviour is characteristic of face centred cubic HEA's with low stacking fault energy.

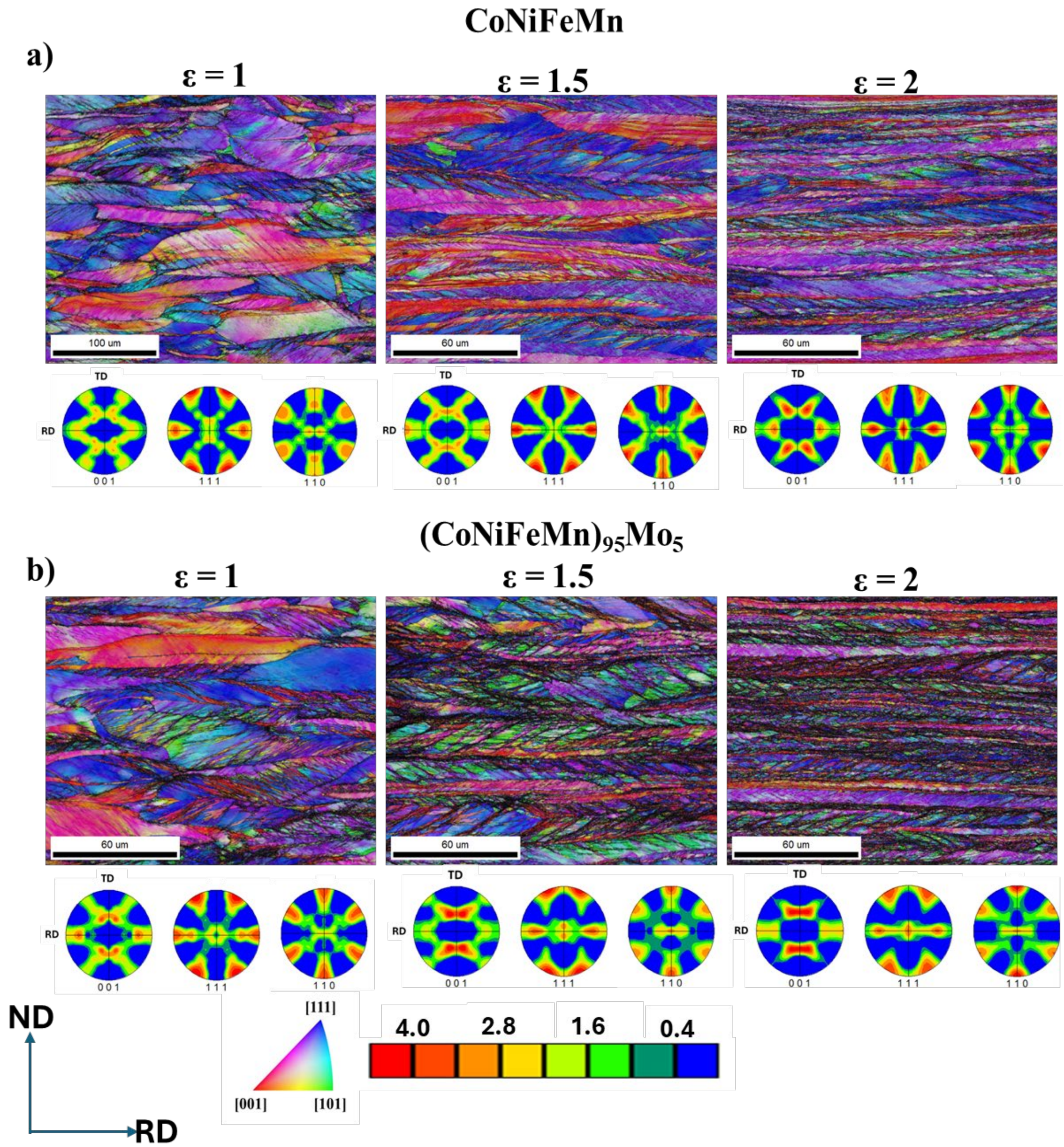

Fig. 3. Inverse pole figure maps and pole figures for a) CoNiFeMn and b) $(CoNiFeMn)_{95}Mo_5$ alloys with different strain level.

The cold rolled materials were subjected to recrystallization annealing. The annealing temperatures and holding times were selected based on our previous work [15]. In the CoNiFeMn alloy, the formation of a heterogeneous microstructure was observed at an annealing temperature of 675 °C with a holding time of 600 s. For the $(CoNiFeMn)_{95}Mo_5$ alloy, a heterogeneous microstructure developed after annealing at 800 °C for 1800 s (Fig. 4a, b). The resulting microstructure is characterized by bands of coarse grains aligned along the rolling direction, surrounded by regions of finer grains. In addition, in the $(CoNiFeMn)_{95}Mo_5$ alloy, molybdenum-rich precipitates of the intermetallic μ phase with a trigonal crystal structure were observed (Fig. 4c). This phase precipitates during recrystallization annealing[15]. The formation of the heterogeneous microstructure was observed exclusively at a true strain of ε = 2.

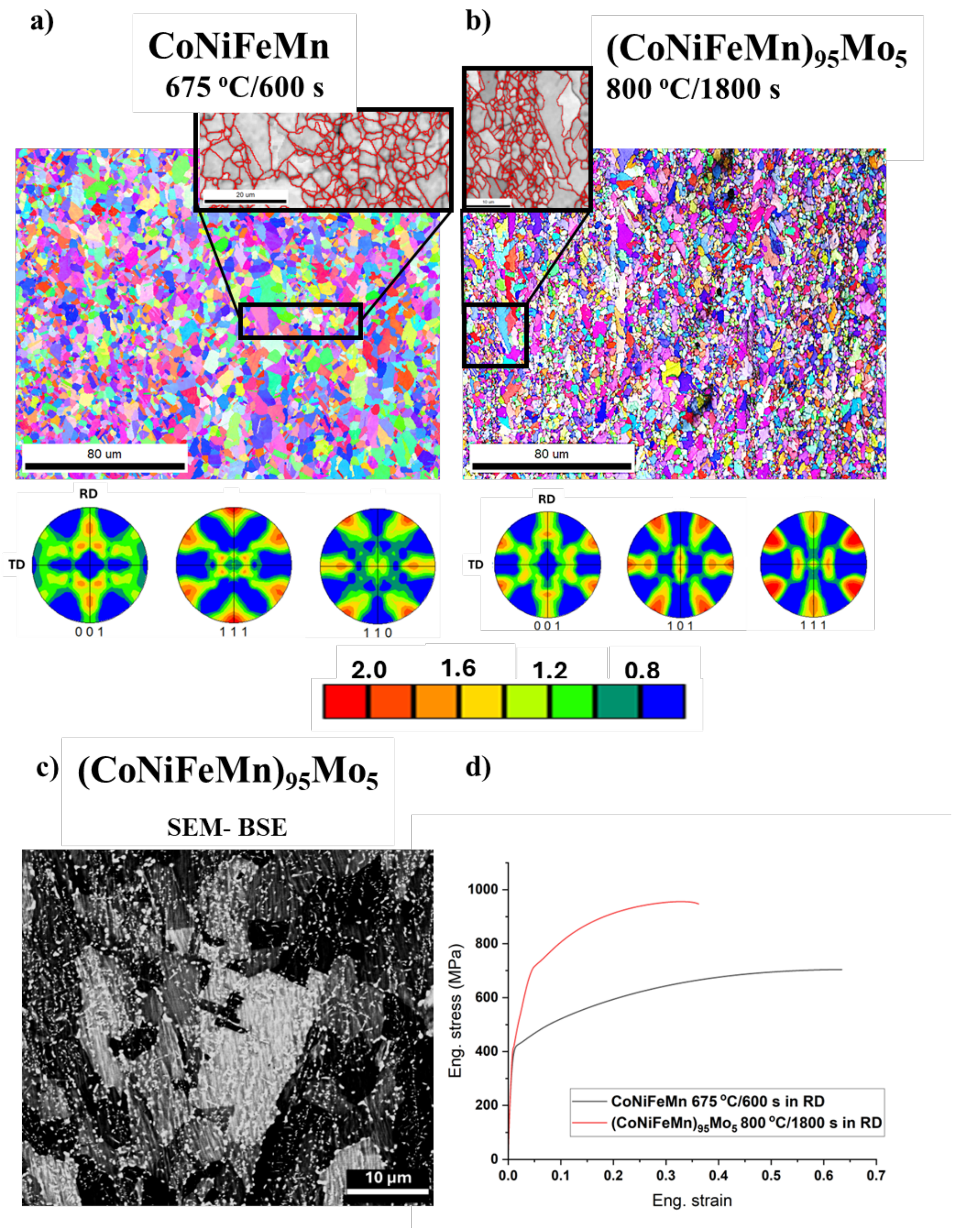

Fig. 4. Inverse pole figure images for annealing sample for CoNiFeMn 675 °C/600 s and CoNiFeMn)$_{95}$Mo$_5$ 800 °C/1800 s with pole figures c) SEM-BSE image for (CoNiFeMn)$_{95}$Mo$_5$ alloy and d) tensile test curves.

The presented pole figures indicate that the materials subjected to static recrystallization remain characterized by a strong crystallographic texture. In both cases, the texture is composed predominantly of the Goss {110}⟨001⟩, A {110}⟨111⟩, F {111}⟨112⟩, and cube {001}⟨100⟩ components. The presence of these texture components is expected to significantly influence the drawability of the investigated materials as well as the operative deformation mechanisms.

Fig. 4d presents the mechanical properties of the alloys selected for the deep-drawing test. The (CoNiFeMn)$_{95}$Mo$_5$ material exhibits significantly higher strength due to the presence of μ-phase precipitates as well as a stronger solid solution strengthening effect. However, its ductility is lower than that of CoNiFeMn. The yield strength, $\sigma_{0.2}$, was estimated at 398 ± 5 MPa for the CoNiFeMn alloy and 545 ± 10 MPa for the Mo-containing alloy.

The annealed materials were subsequently subjected to the Erichsen cupping test (Fig. 5).

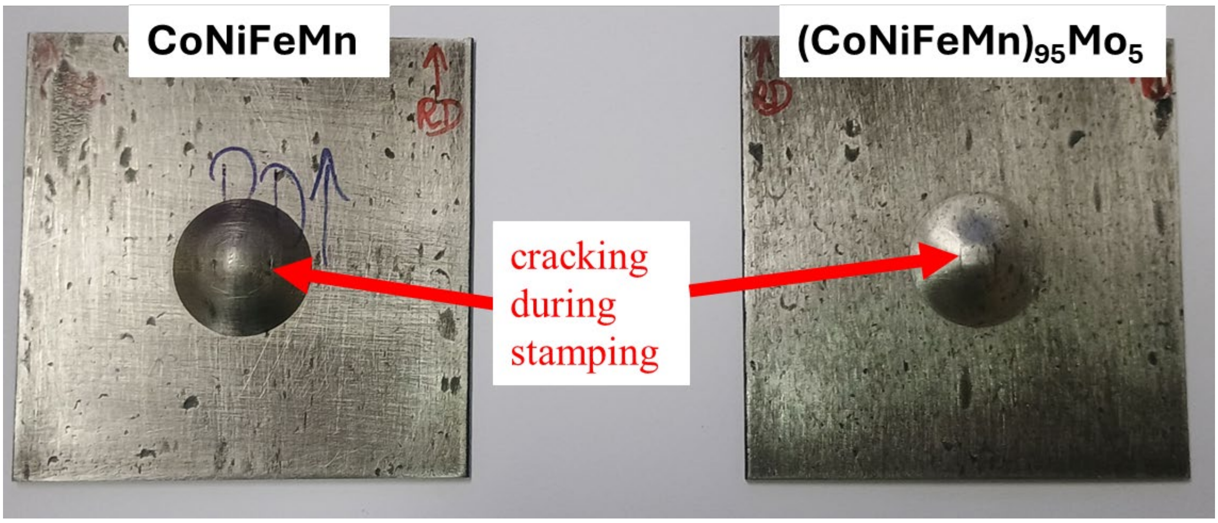

Fig. 5. Samples after Erichsen test.

The Erichsen cupping tests were conducted to evaluate the drawability of the investigated alloys. The CoNiFeMn alloy exhibited an average indentation depth (IE21) of 8.6 mm, indicating good formability under the applied test conditions. In contrast, the $(CoNiFeMn)_{95}Mo_5$ alloy showed a significantly lower average IE21 value of 4.8 mm. This reduction in formability can be primarily attributed to the decreased ductility of the Mo-containing alloy, which is associated with the presence of molybdenum-rich intermetallic μ phase precipitates. These precipitates act as stress concentrators and hinder plastic flow, thereby limiting the material's ability to undergo uniform deformation during deep drawing.

Summary

This study presents a method for producing a heterogeneous microstructure in the $(CoNiFeMn)_{1-x}Mo_x$ alloy system. Two alloys, CoNiFeMn and $(CoNiFeMn)_{95}Mo_5$, were investigated. The materials were subjected to cold rolling followed by recrystallization annealing.

- The formation of a heterogeneous microstructure was successfully achieved in the materials deformed to a true strain of $\varepsilon = 2$. In the CoNiFeMn alloy, the heterogeneous microstructure is characterized by bands of coarse grains surrounded by finer grains, whereas in the $(CoNiFeMn)_{95}Mo_5$ alloy, the presence of molybdenum-rich μ phase precipitates further contributes to the heterogeneity.
- Formability tests demonstrated that the CoNiFeMn alloy exhibits good drawability, whereas the addition of molybdenum significantly reduces formability due to the presence of μ-phase precipitates. Nevertheless, the investigated materials show potential for deep drawing applications, depending on the required balance between strength and ductility

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