Article

Texture Evolution and Plastic Deformation Mechanism of Cold-Drawn Co-Cr-Ni-Mo Alloy

Hanyuan Liu 1,2, Rui Hu 1,*, Xupeng Xia 1,2 and Sen Yu 2,*

1 State Key Laboratory of Solidification Processing, Northwest Polytechnical University, Xi’an 710072, China
2 Shaanxi Key Laboratory of Biomedical Metal Materials, Northwest Institute for Nonferrous Metal Research, Xi’an 710016, China
* Correspondence: rhu@nwpu.edu.cn; ninbrc@163.com (S.Y.); Tel.: +86-29-88492172 (R.H.); +86-29-86231103 (S.Y.)

Abstract: The plastic deformation behavior and mechanisms of Co-Cr-Ni-Mo alloy were investigated. The wires were subjected to different reductions using a multi-pass drawing approach and the resulting microstructures were characterized by EBSD and TEM. It was found that the alloy cold-drawn from surface to center exhibited non-uniform radial strain, with decreasing strain from surface to center. As the strain increased, the transverse texture of the alloy evolved from the initial bimodal texture consisting of strong [100]<110> and weak [110]<001> components to bimodal texture with [110]<233> and [112]<111> components, with significant twinning and mirror orientation between twin and matrix. The longitudinal texture evolution of the alloy mainly occurred on the α-fiber line, and ultimately did not form a significant texture due to grain elongation and crystal rotation. The plastic deformation mechanism of the Co-Cr-Ni-Mo alloy was dominated by dislocation slip at lower strain levels, which gradually transitioned to a combination of dislocation slip and twinning at higher strain levels. The deformation twins were typically distributed in high-density dislocation regions, and the twin boundaries transformed into high-angle sub-grain boundaries, hindering the extension of dislocation slip and deformation twin. With the increase in strain, work hardening results in a significant increase in strength and microhardness.

Keywords: Co-Cr-Ni-Mo alloy; cold-drawing; texture evolution; deformation twins

1. Introduction

Co-Cr-Ni-Mo alloy (MP35N) is a multi-component alloy developed by SPS Technologies Inc. (Jenkintown, Pennsylvania, USA), which has a composition of 35% Co, 35% Ni, 20% Cr, and 10% Mo. It has a similar composition to some non-isotomic medium-entropy alloys and a lower stacking fault energy (SFE), which makes it more prone to twinning compared to high-entropy alloys [1]. This alloy exhibits remarkable strength and ductility, and excellent fracture resistance and corrosion properties [2–4], which is the ideal material for the medical industry such as cardiac pacemaker, defibrillator and nerve stimulator. For the required application, the material needs to be processed into thin fine wires.

In the last 40 years [5–7], most research had focused on the influence of drawing factors and their combinations on the mechanics of plastic deformation by rods or wire drawing. It is well known that plastic deformation has a significant effect on the microstructure, mechanical properties, strain hardening properties, tensile properties, resistance properties, and fatigue properties of the wire [8–13]. MP35N alloy wires need to be manufactured by the cold wire-drawing process which involves a reduction in the cross-section of the wire or diameter, by passing it through different diameter dies. Wire-drawing technology has attracted much attention for its high controllability and high precision in the continuous processing of small-size wires at room temperature, and has been proven to have a significant impact on mechanical properties [14]. This process involves increasing the
strength and ductility reduction in the wires by increasing cold work (CW%) [15,16]. The leads were assembled from thin diameter wires to form a larger diameter coil or cable, which was then post-processed to form various designs depending on their application. However, the dimensional accuracy and properties of the wires continue to be insufficient, and fatigued break occurs frequently in the actual machining process. The dimensional accuracy and surface finish of the wires are usually determined by the cold-drawing texture. In addition, the texture introduced by cold drawing can also affect the mechanical performance of the wires. Therefore, it is necessary to further study the texture evolution behavior of the wire in addition to considering the influence factors of conventional deformation. It is well known that texture is a microstructure phenomenon [10], and a strong understanding of the machining method and its interrelationship with internal and external microstructure parameters and its contribution to the deformation mechanism has been obtained through EBSD and TEM analysis.

Due to high Co content, MP35N alloy has low fault energy, so deformation twins are easily formed during deformation. The deformation of micro-twins will greatly increase the strength of the alloy, and the influence of texture on the strain hardening effect of the alloy during deformation cannot be ignored, which will affect the further processing of the alloy. Exploring the effect of deformation twins on the mechanical properties of MP35N alloy can provide a theoretical basis for further improving the alloy processing technology and expanding the application field of the alloy. So, the purpose of the current study is to characterize the texture evolution during plastic deformation of MP35N alloy, investigate the influence of twinning on texture, and explore the plastic deformation mechanism of the alloy. The research is beneficial for the manufacture of MP35N alloy wires with better performance.

2. Experimental

The MP35N alloy used in this study is a quaternary alloy composed mainly of Co and Ni, with the addition of Cr and Mo elements (Table 1). The Φ15 mm alloy rod obtained was subjected to hot rotary forging at 1000–1100 °C, and after four passes, was forged into a Φ5 × 100 mm fine rod. The uniform forged microstructure was obtained by annealing at 950 °C for 1 h to eliminate internal stress, serving as the initial cold-drawn sample. The cold-drawing rate of the alloy rod was 0.7–0.8 m/min, and it was finally cold-drawn to a diameter of Φ3.4 mm. The amount of deformation during cold drawing was calculated by the following formula [17]:

\[ \varepsilon = \ln \frac{S_0}{S} = 2 \ln \frac{d_0}{d} \]  

where: \( \varepsilon \) is the strain, \( S_0 \) is the cross-sectional area before cold drawing, \( S \) is the cross-sectional area after cold drawing, \( d_0 \) is the diameter before cold drawing, and \( d \) is the diameter after cold drawing.

<table>
<thead>
<tr>
<th>Element</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Fe</th>
<th>Mn</th>
<th>Si</th>
<th>Co</th>
</tr>
</thead>
<tbody>
<tr>
<td>Content</td>
<td>36.13</td>
<td>19.53</td>
<td>9.81</td>
<td>0.42</td>
<td>0.05</td>
<td>0.09</td>
<td>Bal.</td>
</tr>
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The specimens for optical microscopy analysis were first polished with sandpaper and then polished with a 50 nm silica polishing fluid for 20 min. After polishing, surface etching was carried out using modified aqua regia, which was a concentrated mixture of hydrochloric acid and nitric acid in a 2:1 ratio, for about 30 s.

The microstructural orientation features of the deformed specimens were analyzed using electron backscatter diffraction (EBSD). The preparation of EBSD samples involved mechanical polishing like metallographic samples. After mechanical polishing, the
specimens were subjected to vibratory polishing using a silica polishing fluid at a frequency of 62 Hz for 8 h. The vibratory-polished specimens were cleaned in an ultrasonic cleaner, wiped with cotton soaked in flowing water in the direction of the water flow, and then rinsed with anhydrous ethanol and blown dry to complete the EBSD sample preparation. The EBSD analysis was performed using a ZEISS Sigma 300 field-emission scanning electron microscope, (Carl Zeiss AG, USA) and the cross-sections of the deformed samples analyzed included longitudinal and transverse sections, as shown in Figure 1.

In EBSD data analysis, to quantitatively analyze the texture components, the distribution of the orientation distribution function (ODF) in the orientation space composed of Euler angles ($\varphi_1, \Phi, \varphi_2$) is an important basis for determining the texture. The orientation space is composed of three Euler angles with a value range of 0°–90°, and most textures are distributed on the $\varphi_2 = 45^\circ$ section and the 90° section. Therefore, this paper mainly analyzes the texture components on two sections. Figure 2 shows the texture distribution on the two sections, and the analysis data are processed using ATEX V2.08 [18] and Channel 5 V2007 software.

![Figure 1. EBSD analysis section and sample coordinate system (a) longitudinal section; (b) cross-section.](image1)

![Figure 2. Texture distribution of different sections in orientation space (a) $\varphi_2 = 45^\circ$ section; (b) $\varphi_2 = 90^\circ$ section.](image2)

The preparation of TEM samples requires cutting the sample and grinding it on sandpaper to a thickness of less than 30 µm with a flat surface, followed by ion thinning until
enough thin regions appear. The ion thinning equipment used is the Gatan ion thinning instrument model 695 PIPS COOL. The TEM data is analyzed using Digital Micrograph software. The transmission analysis was performed using a field-emission transmission electron microscope FEI Talos F200X (Thermo Fisher Scientific, USA), which can be used for both conventional TEM observations and high-resolution imaging (HRTEM) for analyzing samples at smaller scales.

3. Result
3.1. MP35N Microstructure

The initial microstructure of the alloy was obtained through heat rotary forging and annealing. According to the literature [19], precipitates were observed in some heat-treated MP35N alloys. Therefore, XRD analysis of the alloy was performed, and Figure 3 shows the XRD patterns of the alloy under different processing conditions. The indices corresponding to each diffraction peak indicate that the MP35N alloy has an FCC structure with a lattice constant of 3.58 Å, which is close to that of metallic nickel (3.52 Å), suggesting that it is a solid solution formed by other elements dissolved in the nickel lattice. This indicates that the initial and plastic deformation microstructure of the MP35N alloy is a single-phase structure without precipitates [11].

![Figure 3. XRD patterns of MP35N alloys in different treatment conditions.](image)

Figure 4 shows the EBSD image of the cross-section of the initial MP35N alloy. Figure 4a is the orientation map of the microstructure, which indicates that the initial grains are equiaxed with a size range of 20–30 µm, and there are many annealing twins inside the grains. The width of the twin lamellae mostly ranges from 1 µm to 4 µm and can reach up to 7 µm. Figure 4b is the IPF image parallel to the rolling direction (RD), showing that the grains parallel to the RD direction have a dominant [110] orientation (color green), while the other orientations are close to a random distribution. It is worth noting that most of the twins have a significant orientation difference with the matrix, while in the process of forming some annealing twins, when the twinning direction is the same as the RD direction, the twin orientation will be the same as the matrix, and no orientation difference will be formed between them. Figure 4c shows the distribution of grain boundaries (GBs) and twin boundaries (TBs) in the initial microstructure. Most of the TBs extend from the grain boundaries to the interior of the grains and even penetrate through the entire grains, but they do not cross the grain boundaries. Figure 4d is the map of the adjacent grain orientation difference, showing a significant orientation difference at the grain boundary and a consistent orientation inside the grains, which is consistent with Figure 4b.

The orientation distribution function (ODF) can more clearly display the microstructure orientation. Figure 5 shows that a strong [100]<110> texture and a weak [110]<001>
texture were formed in the initial microstructure, which are common recrystallization textures [20]. In Figure 5b, the area around the \{100\}<110> texture does not have the highest density, but instead forms a bimodal structure. However, compared with other areas, the density of orientations in this area is significantly enhanced.

![Figure 4](image1.png)

**Figure 4.** Initial organization EBSD chart (a) Euler angle image; (b) IPF diagram in RD direction; (c) grain boundary (GB) and twin boundary (TB); (d) misorientation map.

![Figure 5](image2.png)

**Figure 5.** Initial texture Euler space section (a) \(\phi_2 = 45^\circ\); (b) \(\phi_2 = 90^\circ\).

3.2. Evolution of Transverse Texture in Cold-Drawn MP35N Alloy

During the cold-drawing process, the alloy grains rotate with deformation, and the degree of rotation or elongation of the grains towards a certain direction increases with the increase in deformation. Figure 6 shows the imaging of the microstructure of alloys with different deformation amounts parallel to the RD direction. Compared with the initial structure, the orientation of the deformed structure has undergone significant changes. When the strain is 0.21, the proportion of [111]/RD orientation is 23.83%, and the proportion of [001]/RD orientation is 10.72%. Compared with the initial structure, the number of grains with these two orientations increases, while the grains with [101]/RD orientation decrease. The region pointed by the arrow in Figure 6a also shows significant orientation changes within a single grain, indicating the existence of inhomogeneous
deformation within a single crystal. Figure 6b shows the orientation of the texture at a strain of 0.44, where most of the texture orientation is [111]//RD, accounting for 49.64%, and the proportion of [001]//RD orientation is 16.91%, which decreases. That is, a strong [111]//RD and a weak [001]//RD texture are formed. Although the strain increases by 0.33 from 0.44 to 0.77, the texture does not change significantly, and the proportion of [111]//RD orientation is 48.59%, and the proportion of [001]//RD orientation is 15.90%, which is slightly lower than that of the 0.44 strain, indicating that in further deformation process, the crystals no longer rotate but mainly elongate, resulting in smaller grain size on the cross section, increased fragmentation in local areas, and more complex crystal orientations in the fragmented area.

Figure 6 shows that a lot of twin crystals appear in the deformed texture. The representation of twin crystals is usually performed using the heavy and position lattice models, which are mainly used to represent large-angle interfaces. For a certain crystal, rotating a certain angle around a certain crystal axis obtains another crystal with a different orientation. When they are extended to each other, some atoms overlap with each other periodically. These overlapping atoms form a new lattice, called a coincident position lattice. The ratio of overlapping position atoms to the original lattice is denoted as “coincident position density” and is represented by 1/Σ. The coincident position density of the heavy position lattice obtained by rotating around the <111> crystal direction in the face-centered cubic crystal by 60° is one-third, also known as a twin boundary. In EBSD data, the orientation difference between the two crystals can be calculated to determine whether it is a twin boundary. The orientation difference between [111]//RD and [001]//RD orientations calculated in Figure 6b,c indicates that they are 60°, that is, the matrix orientation is [111]//RD, which is the main component of the texture, while the twin orientation is [001]//RD, which is the minor component of the texture. This indicates that the twin and the matrix have orientation differentiation in the parallel RD direction. If the twin orientation is <001>, the matrix orientation is <111>, and the twin plane normal is <111>, the angle between the twin orientation and the twin plane normal is 54.73°, and the angle between the matrix orientation and the twin plane normal is 70.52°. The crystal orientation angle is calculated by the following formula:

$$\cos \theta = \frac{h_1h_2 + k_1k_2 + l_1l_2}{\sqrt{h_1^2 + k_1^2 + l_1^2} \times \sqrt{h_2^2 + k_2^2 + l_2^2}}$$

(2)

where h1, k1 and l1 are one crystallographic index<hk1l>, and h2, k2 and l2 are another crystallographic index<hk2l>; θ is the included angle of two crystal directions.

As the two specific orientations are nearly perpendicular to the twin plane normal, when the matrix orientation tends towards [111]//RD, due to the overall rotational nature of the crystal, the annealing twinned grain orientation will tend towards [001]//RD. In fact, a mirror texture is formed. Taylor’s crystal plasticity theory shows that [21], during the plastic deformation process, the crystal will undergo rotation, and the ease of rotation is related to the deformation direction. That is, relative to the RD direction, the smaller the Schmid factor of a certain orientation, the easier it is to rotate to a direction parallel to RD. In this alloy, the twin and matrix are highly symmetric, resulting in a significant mirror texture.
Figure 6. Transverse reverse pole image of alloys with different strains (a) 0.21; (b) 0.44; (c) 0.77; (d) Reference orientation color card.

To further analyze the deformation texture of cold-drawn MP35N alloy, an ODF map was generated as shown in Figure 7. At a strain of 0.21, a weak [112]<111>Copper texture was observed in Figure 7a, while the highest orientation density region tended towards a [110]<233>P texture, corresponding to the orientation density on the α orientation line in Figure 7d. A double texture component of [110]<233>+[112]<111> was formed in the structure with a strain of 0.44, which was greatly enhanced compared to the texture at a deformation of 0.21. As the strain was further increased to 0.77, the texture component remained unchanged, but the P texture weakened and the Copper texture strengthened, with a weak [110]<001>Cube texture appearing. Unlike the previous case where the main orientations of the structure were [111]//RD and [001]//RD after a deformation of 0.21, the texture orientations in the three-dimensional orientation distribution function were spatial, while the orientations in the orientation imaging map were only parallel to the RD direction. Therefore, the ODF map shows the actual texture orientations.

Figure 8 shows the orientation density of the orientation lines on two sections with $\varphi_2 = 45^\circ$ and $\varphi_2 = 90^\circ$ in Euler space. The content of each texture was quantitatively represented. Figure 8a shows the orientation line with $\varphi_2 = 45^\circ$ and $\varphi_1 = 90^\circ$ in Figure 7a–c, where $\Phi$ ranges from 0° to 90°. As the strain increased, the Copper texture gradually strengthened, and after a strain of 0.44, the growth rate of its orientation density slowed down, consistent with the results shown in the orientation imaging map. The texture extension range gradually increased, and the angle corresponding to the maximum orientation density gradually moved towards $\Phi$ decreasing direction, indicating that the Copper texture gradually strengthened, while other orientations such as the [111]<112>Brass texture [22] showed only slight changes in orientation density at different strains and were not strong enough to form significant textures. Figure 8b shows the orientation density of the $\alpha$ orientation line in Figure 7d–f, which is a set of textures with {011} plane parallel to the rolling plane in the deformed structure. The P texture first increased and then decreased as the strain increased, while the Brass texture gradually weakened. In addition, other texture orientations were relatively weak.
During the deformation process, the evolution of the transverse texture was from the strong [100]<110> and weak [110]<001> initial textures of recrystallization to the [110]<233> and [112]<111> textures. As the strain increased, both texture components were greatly strengthened and then differentiated, with the [110]<233> texture weakening and evolving into [112]<111> and [100]<001> textures.

Figure 7. ODF cross-section of cold-drawn structure with different strain (a) $\epsilon = 0.21$, $\phi_2 = 45^\circ$; (b) $\epsilon = 0.44$, $\phi_2 = 45^\circ$; (c) $\epsilon = 0.77$, $\phi_2 = 45^\circ$; (d) $\epsilon = 0.21$, $\phi_2 = 90^\circ$; (e) $\epsilon = 0.44$, $\phi_2 = 90^\circ$; and (f) $\epsilon = 0.77$, $\phi_2 = 90^\circ$.

Figure 8. Distribution density of orientation line (a) $\phi_2 = 45^\circ$, $\phi_1 = 90^\circ$, $\Phi$ is $0^\circ$–$90^\circ$; (b) $\phi_2 = 90^\circ$, $\alpha$ fiber.
3.3. Longitudinal Texture Evolution of Cold-Drawn MP35N Alloy

In the cold-drawn microstructure, the longitudinal tensile stress dominates, leading to mainly elongation between grains. Figure 9 shows the Euler angle imaging of the longitudinal microstructure, with the RD direction being the cold-drawing direction. At a strain of 0.21, Figure 9a shows no significant changes in the grains along the cold-drawing direction, with the microstructure maintaining the characteristics of equiaxed grains. In Figure 9b, as the strain increases, grains break and elongate along the cold-drawing direction, with some annealing twins bending and the aspect ratio of the grains increasing, indicating coordinated deformation between grains along the cold-drawing direction. Figure 9c shows that when the strain reaches 0.77, the grains are completely elongated, and most of the grain boundaries are parallel to the RD direction, forming a deformation band along the RD direction.

In the plastic deformation process of polycrystal, the grain is mainly subjected to tensile stress in the cold-drawing direction, and the Schmidt factor in this direction is small, which is easier to meet the multi-slip condition, and thus the grain elongates along the cold-drawing direction.

Compared with the transverse texture, the longitudinal texture is not strongly oriented partly because the grain is subjected to the radial shear stress and the RD direction normal stress, that is, the compressive stress, resulting in the longitudinal deformation of the grain during cold drawing, and the grain is easy to rotate only in the soft orientation with large Schmid factor. The grain rotation resistance is very large, and the orientation unity will be enhanced only when the hard oriented grain begin to rotate and the grain length axis ratio increases under greater strain. On the other hand, the crystal is easy to rotate in the cross section under normal stress, and the hard oriented grain rotates under small stress, thus forming a strong texture.

A large number of immovable dislocations, shear bands, and twin boundaries introduced in the cold-drawing process hinder the dislocation movement, thus it can improve the strength and affect the plasticity of the alloy. As a result, the strength of the alloy increases while the ductility decreases.

![Figure 9](image_url)

**Figure 9.** Longitudinal Euler angle imaging of alloys with different strains: (a) \(\varepsilon = 0.21\); (b) \(\varepsilon = 0.44\); (c) \(\varepsilon = 0.77\).

The ODF (orientation distribution function) map of the longitudinal cold-drawn microstructure is shown in Figure 10. In the cross-section with \(\varphi_2 = 45^\circ\), as shown in Figure 10a–c, the texture of the microstructure exhibits a tendency toward the \(\gamma\)-fiber texture, with gradual strengthening of \([111]<112>\)F and \([111]<110>\)E texture components along the \(\gamma\)-fiber texture. In addition, there is also a trend toward the Copper texture component. Regardless of the variation in the deformation amount, the orientation distribution in Figure 10d–f mainly evolves along the \(\alpha\)-fiber texture, with a weakened \([011]<233>\)Goss texture and a strengthened Brass texture [23,24].
Figure 10. ODF cross-section of cold-drawn structure with different strains: (a) $\varepsilon = 0.21, \varphi_2 = 45^\circ$; (b) $\varepsilon = 0.44, \varphi_2 = 45^\circ$; (c) $\varepsilon = 0.77, \varphi_2 = 45^\circ$; (d) $\varepsilon = 0.21, \varphi_2 = 90^\circ$; (e) $\varepsilon = 0.44, \varphi_2 = 90^\circ$; and (f) $\varepsilon = 0.77, \varphi_2 = 90^\circ$.

3.4. Microstructure Evolution of Cold-Drawn MP35N Alloy

As MP35N alloy belongs to low-SFE alloys, a twinning mechanism occurs in addition to the dislocation slip mechanism during deformation. Due to the limitation of resolution and data characteristics, EBSD analysis cannot represent the deformation micro-twins in the microstructure. In this section, the deformation mechanism of the alloy is discussed by combining metallurgical photographs (OM) and TEM analysis.

During the cold-drawing process, areas with high local stress will meet the twinning nucleation conditions. Figure 11 shows the initial microstructure of the alloy, with few dislocations in the structure. Regions with high dislocation density form a dislocation network, while low-density dislocation regions are present internally. In Figure 11b,c, the width of annealed twins is above 500 nm, and the long and straight boundaries are coherent twin boundaries (CTBs), which are parallel to the [111] plane and maintain a coherent relationship with the matrix. They gradually expand and grow towards both sides. The steps between CTBs are incoherent twin boundaries (ITBs). In the high-resolution mode of Figure 11d (larger view of the red square region in Figure 11c), the width of ITBs is measured to be approximately 5 nm.
After 0.21 deformation, due to the small stress, the twinning in the microstructure remains the annealed twinning. Figure 12a shows that the intensity of the twinning spots in the microstructure is similar to that of the matrix in the electron diffraction pattern, and the dark-field image of the twinning spots in the circle is shown in Figure 12c. Twins are formed on both sides of the matrix, with a twin layer thickness of about 55 nm on one side and a twin width above 1 µm on the other. Figure 12d is a magnified view of the boxed area in Figure 12b, showing a group of dislocations moving towards the twin boundary along direction a and forming a dislocation pile-up on interface 1. The spacing between each dislocation in the pile-up is not uniform and follows a parabolic distribution. According to the principle of virtual work, there is a large stress concentration at the interface, as shown in Equation (3):

\[ \tau = n \tau_0 \]  

where \( \tau_0 \) is the stress field component on the dislocation slip plane, \( \tau \) is the shear stress on the pile-up front, \( n \) is the number of pile-up dislocations.

From Equation (3), it can be seen that there is a significant stress concentration at the interface. In order to relieve this stress, a Frank–Read dislocation source is activated on the other side of the twin boundary, and the dislocation emits towards the matrix along direction b. At another twin boundary 2, the dislocation forms a new pile-up, and then two sets of dislocations slip in opposite directions to the twin plane (11T) about a mirror plane of symmetry. The high-resolution image of the boxed area in Figure 12d is shown in Figure 12e, with region A being the annealed twin boundary. On this interface, the crystal twinned again to form a 50 nm wide micro-twin, with stacking faults being the main feature in region B. The fast Fourier transform (FFT) diffraction pattern of the HRTEM image in the upper right corner is the same as that in Figure 12a, indicating that the twinning plane of the deformed twin is the same as that of the annealed twin, and the twin boundary is also the source of new twin nucleation. This indicates that in smaller deformations, the dislocation slip mechanism is dominant, but the twinning mechanism is also involved.
Figure 12. $\varepsilon = 0.21$ TEM micrograph (a) selected area electron diffraction pattern of crystal belt axis $\langle 011 \rangle$; (b) bright-field image; (c) twin dark-field image; (d) amplification of bright-field image area, a and b are dislocation motion directions; 1 and 2 are twin boundaries; (e) HRTEM images and corresponding FFT diffraction patterns of micro-twin growth front, A and B are the atomic distribution at the twin boundary.

Figure 13 shows the TEM image of the 0.44 strain sample. In the bright-field image, a lot of twinned crystals have a consistent twinning direction and eventually terminate at the grain boundaries. The average width of the twinned crystal layers in Figure 13c is 15 nm, and the gaps between the micro-twins are high-density dislocation clusters, which are distributed non-uniformly. In another region in Figure 13d, the diffraction spots corresponding to high-index crystal planes rotate counter-clockwise as a whole, indicating that the stress is high in this area, causing severe lattice distortion, and the deformed twins are not in complete lattice coherency with the matrix. Moreover, the twinned spots are not perfectly circular but elongated into elliptical shapes, which are the diffraction patterns formed by a lot of stacking faults. These stacking faults can be regarded as extremely thin twinned boundaries in a face-centered cubic crystal, and a full dislocation can be decomposed according to Venables' twinning polar mechanism as follows [17]:

$$\frac{1}{2} < 110 > \rightarrow \frac{1}{6} < 121 > + \frac{1}{6} < 21\bar{1} >$$  \hspace{1cm} (4)

A stacking fault is sandwiched between the two dislocations formed after decomposition. Under stress, if the two dislocations rotate in opposite directions at the node, the width of the stacking fault increases by one close-packed plane per revolution. This causes shear deformation of the whole crystal, which readily promotes the formation of microtwins by the accumulation of a lot of stacking faults. Therefore, in the deformation mechanism of the 0.44 strain alloy, dislocation slip and twinning co-dominate.
Figure 13. $\varepsilon = 0.44$ TEM micrograph (a) selected area electron diffraction pattern of crystal belt axis <011>; (b) bright-field image; (c) twin dark-field image; (d) another area bright-field image and corresponding selected area electron diffraction pattern.

Figure 14 shows the TEM image of the alloy after 0.77 strain deformation. As the strain reaches 0.77, the micro-twinning layers become denser. In Figure 14c, most of the twinning occurs between two solid lines, with a layer width of 20 nm. In bright-field images, the region where twinning occurs is characterized by low-density dislocation regions and terminates near high-density dislocation walls. This is because the twin is formed by the complete decomposition of a dislocation into a twin according to Equation (4), releasing internal stress. If further deformed, the stacking faults in the high-density dislocation region will also expand to form a continuous twinning region. If an existing dislocation line is cut by twinning, the dislocation orientation changes, and its new Burgers vector in the twin may differ from that in the matrix. At this time, the twin interface may cause the dislocations inside the twin to decompose into movable dislocations that can cross the interface. Although this only occurs in areas of localized stress concentration, a lot of dislocations satisfying the conditions have already accumulated in the alloy after 0.77 strain deformation, resulting in the fragmentation of twinning formed in the high-density dislocation region, as shown in Figure 14b,c. Figure 14d is the HRTEM image of the twinning region, and the FFT spectrum in the upper right corner shows that although significant shear deformation occurs in the crystal along the (111) plane, no diffraction spots are formed, but rather a line pattern is observed. This indicates that the region is mainly composed of stacking faults, which will further develop into micro-twins, as discussed earlier. Figure 14e shows the configuration of the annealed twin in the deformed structure, with a width of about 50 nm and filled with high-density dislocations inside the twin, indicating that the dislocations inside the twin are also activated during deformation, but their motion is limited to the twin boundary. The leading edge of the annealed twin is magnified in Figure 14f. On side A of the annealed twin, the CTB interface disappears, and deformation micro-twins are dominant inside the annealed twin. These micro-twins terminate at the original CTB interface, while on side B near the interface,
dislocations are dominant and remain coherent with the matrix. This indicates that during deformation, dislocation proliferation and twinning also occur inside the annealed twin, but due to the twinning shear deformation, these micro-twins and dislocations cannot cross the CTB interface, which has actually become a high-angle sub-grain boundary, hindering dislocation motion and contributing to the high strain hardening rate of the MP35N alloy.

In face-centered cubic crystals, during deformation, dislocations typically glide along the close-packed plane \(\{111\}\) in the direction of slip \(<110>\), and only when the critical shear stress is reached can the dislocation slip. Equation (2) shows that when the normal stress is constant, dislocations are more likely to glide on the “soft orientation” with a larger Schmid factor. Therefore, when the strain is small, only the slip systems with the “soft orientation” in the grains are activated. In each grain, there is only one slip system with the maximum orientation factor, and the observed slip systems are parallel to each other and highly concentrated, forming slip bands visible under a microscope. At this point, a single-system slip occurs. As the strain increases, that is, when \(\sigma\) increases, slip systems with smaller \(m\) values will also reach the critical shear stress and undergo multiple-system slip. Different slip systems will intersect, and the angle between the slip planes can be calculated using Equation (2) as \(70.53^\circ\). Multiple slip systems cause dislocations to intersect and produce cutting steps, which move together with the dislocations, increasing the resistance and thus the strength of the alloy. As the strain further increases, more slip bands appear, causing the crystal to rotate. In alloy of 0.77 strain, previously formed slip bands are bent, but in low-SFE alloys, obstructed dislocations undergoing multiple slip will not intersect, but rather decompose into extended dislocations, generating micro-twins to relax the stress.

Schmidt’s law is as follows:

\[
\tau = \sigma \cos \lambda \cos \phi = m \sigma
\]

(5)

where \(\tau\) is shear stress in sliding direction; \(\sigma\) is normal stress; \(\lambda\) is the angle between \(\sigma\) and slip direction; \(\phi\) is the angle between \(\sigma\) and normal of sliding surface; \(m = \cos \lambda \cos \phi\) — Schmidt factor.

**Figure 14.** \(\epsilon = 0.77\) TEM micrograph (a) selected area electron diffraction pattern of crystal belt axis \(<011>\); (b) bright-field image; (c) twin dark-field image; (d) HRTEM diagram of twin band in
In some low-SFE high-entropy alloys containing Co [25–27], dislocation slip and twinning are the main deformation at room temperature, while martensitic transformation occurs at low-temperature deformation below 173 K, which indicates that martensitic transformation does occur during deformation of multi-component alloys, but the temperature dependence is greater. In the SAED images observed above, except for matrix and twin spots, plastic deformation does not produce martensite diffraction spots, so it can be determined that the cold-drawing mechanism is slip and twinned.

### 3.5. Mechanical Property of Cold-Drawn MP35N Alloy

With the increase in strain, the deformation mechanism of cold-drawn MP35N alloy changes from dislocation slip to twin. The density of dislocation and micro-twins is greatly increased, resulting in a strain hardening effect. Figure 15 shows the variation curve of alloy microhardness with stress variables. The hardness of the alloy increases from the initial 310.9 HV to 435.1 HV when the cold-drawing amount is small, which is due to the large proliferation of dislocation resulting in hardening. It can be seen from the microstructure that both grain boundary and annealing twin boundary may be the source of dislocation, and because of the presence of annealing twins, the interface has a stronger hindering effect on dislocation, so the hardening effect is more significant than that of low strain. When the strain variable reached 0.44, both dislocation and twins proliferated, a large number of slip bands was observed in the metallographic structure, and the microhardness was further enhanced. Compared with 0.44, the strain of 0.77 had a smaller increase in hardness, and a large number of dislocations appeared in the structure, and the deformation mechanism changed to mainly twinning. However, the hardening effect of twins is lower than that of dislocations, and the twin regions are mostly located in high-density dislocation regions, which already exist at the strain variable of 0.44, so the microhardness is only increased by 8.6 HV [28].

![Microhardness of cold drawing with different strains.](image)

Figure 15. Microhardness of cold drawing with different strains.

Tensile properties of alloy samples with different treatment states were tested at room temperature, as shown in Figure 16. Figure 16a shows the tensile stress–strain curves at room temperature of the alloy samples in different treatment states. The plasticity of the initial alloy is the best, the elongation reaches 78%, and the yield strength and tensile strength are 455.17 MPa and 966.7 MPa, respectively, which are also the basis for cold drawing of the alloy. The yield strength and tensile strength of the alloy increase greatly with the increase in the strain variable, while the plasticity decreases greatly. It can be seen from the above that a large number of immovable dislocations, shear bands, and twin boundaries introduced in the cold-drawing process can hinder the dislocation movement,
thus improving the strength and affecting the plasticity of the alloy. As a result, the strength of the alloy increases while the ductility decreases.

As shown in Figure 16, the tensile stress–strain curve of the initial alloy has a yield point of 2.12, and there is an obvious yield point. After a certain strain, there is already a stress in the alloy itself, and such stress increases with the increase in the strain variable, and the dislocation can only be started under greater stress. Therefore, the stress–strain curve of the alloy after cold drawing with a strain variable of 0.22 has not activated the new dislocation after reaching yield, and still maintains the characteristics of the approximate elastic stage. It does not enter the plastic deformation stage until about 1100 MPa, when the movable dislocation and dislocation source are activated in large quantities. However, its strength only increases by tens of MPa before it breaks. If the engineering stress is transformed into true stress, the true stress increases greatly. This characteristic is similar to that of alloys with general stress–strain curves after unloading and reloading after reaching a certain shape, where the yield strength increases and the plasticity decreases. This feature is more obvious in the stress–strain curves of alloys after cold drawing with strain variable of 0.44 and 0.77. After cold drawing with strain variable of 0.77, the plasticity of alloys is almost the same as that of alloys with strain variable of 0.44, but the tensile strength is increased by 261 MPa, mainly because the deformation mechanism has changed into a twinned one. Twinning shear requires greater stress to proceed, while plasticity remains the same as 0.44 due to twinning.

Figure 16b is a local magnification diagram of the yield section of the alloy. Despite the difference in cold-drawing amount, obvious stress relaxation occurs when the alloy enters the plastic deformation stage, and all of them occur at a strain variable of about 0.016. The greatest stress relaxation is the alloy with a strain variable of 0.77 after cold drawing, up to 40 MPa. After stress relaxation, the alloy evolves along the same trend of growth as the original curve, which may be related to the start-up of the twin system. The dislocation density in the initial alloy is very low, so some plastic deformation has occurred before the stress relaxation, resulting in dislocation proliferation. The local stress increase causes the twin system to start and form micro-twins. This leads to stress relaxation. However, there are a lot of high-density dislocation areas in the cold-drawn alloy, which will also promote the formation of micro-twins after reaching a certain stress, resulting in stress relaxation [29].
4. Conclusions

In this paper, the microstructure evolution on the mechanical properties of MP35N alloy is analyzed, which provides theoretical guidance for the plastic processing and strengthening and toughening of MP35N alloy. The main conclusions are as follows:

(1) The initial structure of MP35N alloy is equiaxed. With the increase in strain, the transverse texture of the alloy changes from the dual texture components of the initial structure (100)<110> strong texture and (110)<001> weak texture to the dual texture components of (110)<233> and (112)<111>, and there is a significant mirror orientation of twins and matrix. In contrast, the longitudinal texture evolution of the alloy is mainly on the α orientation line, no significant texture is formed due to grain elongation and crystal rotation.

(2) The plastic deformation mechanism of MP35N alloy is mainly dislocation slip at first, and with the increase in strain, it changes to dislocation and twinning. The deformation twins produced by twinning are usually distributed in the high-density dislocation region. The same dislocation slip and twinning occur in the annealing twins and the matrix, and the twin boundary is transformed into high-angle sub-grain boundary, which becomes the obstacle of dislocation slip and deformation micro-twin expansion.

(3) Cold drawing results in strain hardening of the alloy. The microhardness of the alloy after cold drawing at a strain variable of 0.77 reaches 531.9 HV, which is 220 HV higher than the initial state. The maximum yield strength and tensile strength of the cold-drawn alloy are 1351 MPa and 1758 MPa, respectively. The operation of the twin system causes obvious stress relaxation in the stress–strain curve.

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