# ABNORMAL PLASTIC BEHAVIOR OF FINE GRAIN MP35N ALLOY DURING ROOM TEMPERATURE TENSILE TESTING

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**Abstract** In this paper, results of an investigation on the strain hardening responses of superalloy MP35N with two average grain sizes of 38 and 1 μm, during room temperature tensile testing are reported. The microstructural evolution of the deformed samples was studied using optical and transmission electron microscopy (TEM) techniques. The strain hardening behavior of the 38 μm material was rather similar to that previously reported for low stacking fault energy FCC alloys. The plastic behavior of the fine-grained material, however, was unexpected. In the strain range of 0.1-0.4, the work hardening rate of the fine grain size sample was evidently lower than that of the large grain size material. Based on the TEM results obtained in this study, it is suggested that the unusual strain hardening response of MP35N alloy of 1 μm grain size is related to the difficulty of formation of deformation twins in these samples. Results obtained in this study further support the effectiveness of deformation twins in hindering crystallographic slip in low SFE FCC polycrystals and the need to develop new plasticity models to include the role of deformation twinning in these materials.

**Key Words** Strain Hardening, Low SFE, MP35N, Deformation Twinning

**چکیده** در این مقاله نتایج تحقیقات انجام شده بر رفتار کار سختی سوپر آلیاژ MP35N در دمای محیط و در دو دانه بندی یک میکرون و ۳۸ میکرون ارائه شده است. تغییرات ریز ساختاری نمونه های تغییر شکل داده شده توسط میکروسکوپ نوری و میکروسکوپ الکترونی عبوری (TEM) بررسی گردید. نتایج بدست آمده نشان دادند که رفتار کار سختی آلیاژ با دانه بندی ۳۸ میکرون مشابه رفتار گزارش شده در مورد آلیاژهای FCC با انرژی نقص چیده شدن (SFE) کم می‌باشد. اما رفتار نمونه های دانه ریز کاملاً غیر منتظره بود. بویژه در محدودهٔ کرنشهای ۰/۱ تا ۰/۴، سرعت کار سختی نمونه های دانه ریز، به مقدار قابل توجُهی کمتر از نمونه های دانه درشت اندازه گیری گردید. بر مبنای نتایج بدست آمده از مطالعات TEM در این تحقیق، پیشنهاد شده است که این رفتار غیر عادی در نمونه های دانه ریز مرتبط با دشواری در تشکیل دو قلوئی های مکانیکی می‌باشد. نتایج بدست آمده در این تحقیق تأثیر دوقلویی های مکانیکی در جلوگیری از لغزش نابجایی را تأیید کرده و نیاز به ارائهٔ مدلهای پلاستیسیته ای که در بر گیرندهٔ نقش دو قلوهای مکانیکی در کار سختی مواد باشد را مورد تأکید قرار می‌دهد.

## 1. INTRODUCTION

High strength and high resistance to deformation and fracture are essential requirements for modern engineering materials. Forming processes, however, require sufficient ductility to allow large deformations to be achieved with a reasonable force. Knowledge of strength and plasticity of materials is therefore essential for optimizing the design and manufacturing processes. On the other hand, it is well established that properties of materials are structure-sensitive. Understanding the mechanical behavior of a material in terms of its structure is thus an important step in utilizing its full potentials for engineering applications by improving or controlling its properties. This has been a major task in modern technologies in order to provide the necessary knowledge and background for further technological progress.

MP35N (35%Ni-35%Co-20%Cr-10%Mo) is a wrought superalloy extensively used in aerospace, marine and petrochemical industries. When fully annealed, the alloy is a single-phase material with FCC structure. Previous studies [1-4] on the strengthening mechanisms of MP35N have shown that the alloy with 30-40 μm grain size gains its strength through work hardening and a second mechanism known as secondary hardening. These strengthening mechanisms have been recently investigated [5,6]. The striking feature of MP35N alloy with 30-40 μm grain size is a four stage work hardening rate at room temperature which is shown to be characteristic of low stacking fault energy (SFE) alloys with comparable grain sizes [5]. It was shown that the high work hardening rate observed in MP35N was due to profuse twinning, implying the effectiveness of twin boundaries in hindering the crystallographic slip [5].

An interesting property of MP35N is the possibility of producing very fine grain sizes simply by heat treating the deformed material [4]. While important aspects of plastic behavior of MP35N alloy with 30-40 μm grain size have been investigated in previous studies, there has been no detailed investigation on the effect of grain size on strain hardening behavior of this alloy. On the other hand, it has been reported that very fine grain sizes may completely inhibit the formation of twins [8,9] and martensite [10]. The main goals of this investigation have been (i) to characterize the strain hardening response of the fine grain size alloy in uniaxial tension and (ii) to study the effects of small grain size on the evolution of microstructure of MP35N alloy during deformation.

## 2. MATERIAL AND EXPERIMENTAL

Raw material used in this investigation was supplied by SPS technologies, Jenkintown USA, in the form of rods with 52 percent cold work. Samples with average grain size of 38 μm were prepared after heat-treating the raw material at 1050 °C for one hour in an inert atmosphere. To obtain a grain size of about 1μm, samples of raw material were heat treated at 870 °C for two hours.

Round tensile samples with diameter of about 6 mm were prepared from MP35N alloy with both grain sizes according to ASTM E8 standard. Tensile tests were performed at a constant true strain rate of 0.001 $(sec)^{-1}$ at room temperature using a computer-controlled servo-hydraulic INSTRON 8500 mechanical testing unit. Raw data were collected as load-displacement and true stress-true strain curves were calculated for both grain sizes up to a plastic strain of about 0.4, well before necking started. Also, for each grain size, strain-hardening response of the alloy was plotted versus true strain.

To investigate the microstructural changes associated with plastic deformation, samples of MP35N with both grain sizes were deformed to varying strain levels in the range of 0.05 to 0.4 in tension. Selected samples with larger grain size were then sectioned parallel to the loading direction, and mechanically ground. The samples were electropolished and electroetched in a solution of 35% $H_2SO_4$ and 65% methanol at 0°C and an operating voltage of 12-15 volts. These samples were then examined using an Olympus PMG3 optical microscope.

For more detailed microstructural examination, a number of TEM samples were prepared from selected samples with 38 and 1 μm grain sizes. Using electric discharge machine (EDM), slices of about 0.4 mm in thickness were cut transverse to the loading direction and were subsequently ground to a thickness of 0.1 mm. Discs of 3 mm in diameter were cut from the thinned samples and TEM foils were prepared by electropolishing these discs in a Fischone twin-jet unit. A solution of 60 ml $H_2SO_4$, 15 ml $H_3PO_4$, and 240 ml methanol was used at 0°C and an operating voltage of 10 volts. A Philips CM200 TEM/STEM operating at 200 kV was used for electron microscopy studies.

## 3. RESULTS

**Tensile Test** Figure 1 shows the true stress-true strain response of annealed MP35N with both grain sizes. The variation of normalized strain hardening rate, $(d\sigma/d\varepsilon)/G$, with true strain for both grain sizes is shown in Figure 2. These figures reveal some striking differences between the work hardening behaviors of 1 μm and 38 μm grain size alloy. Up to a strain of 0.05, a regime of decreasing strain hardening rate identical to stage III (dynamic recovery) of metals of higher SFE exists in the

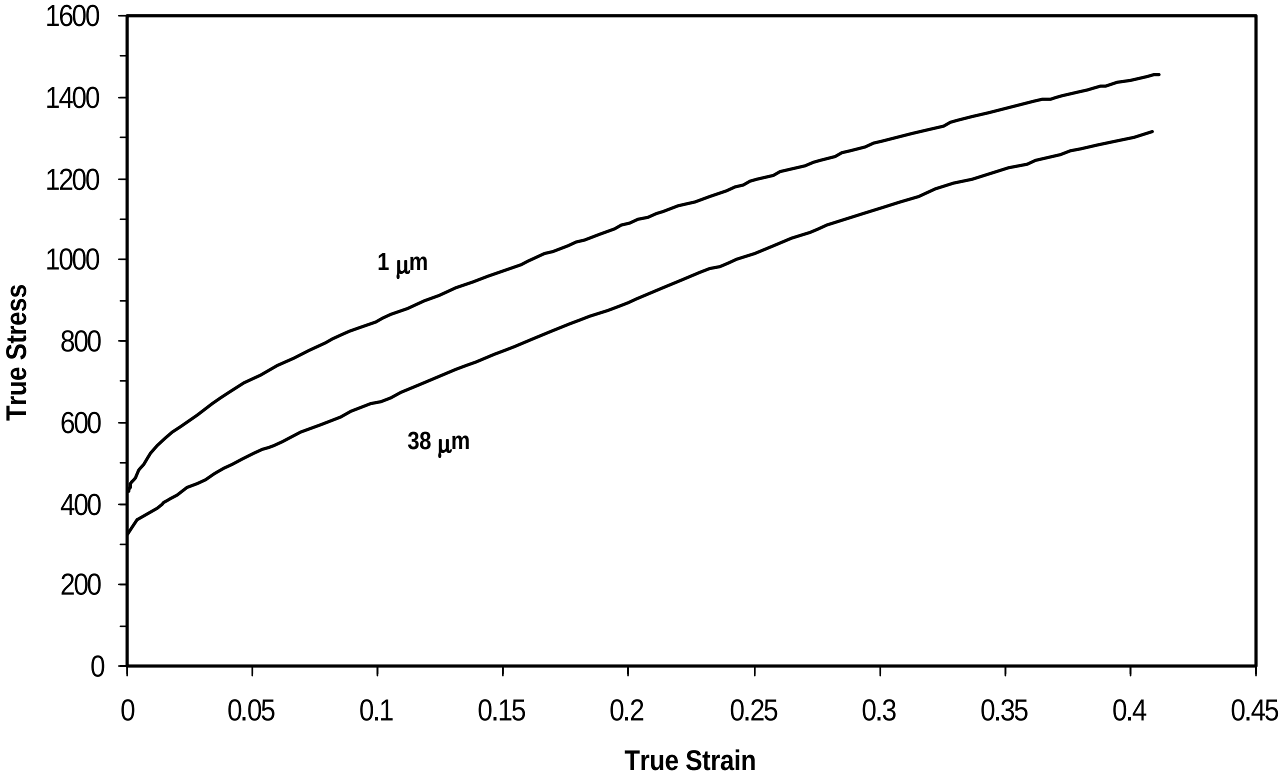



**Figure 2**. Tensile true stress-true strain response of annealed MP35N with two different grain sizes.

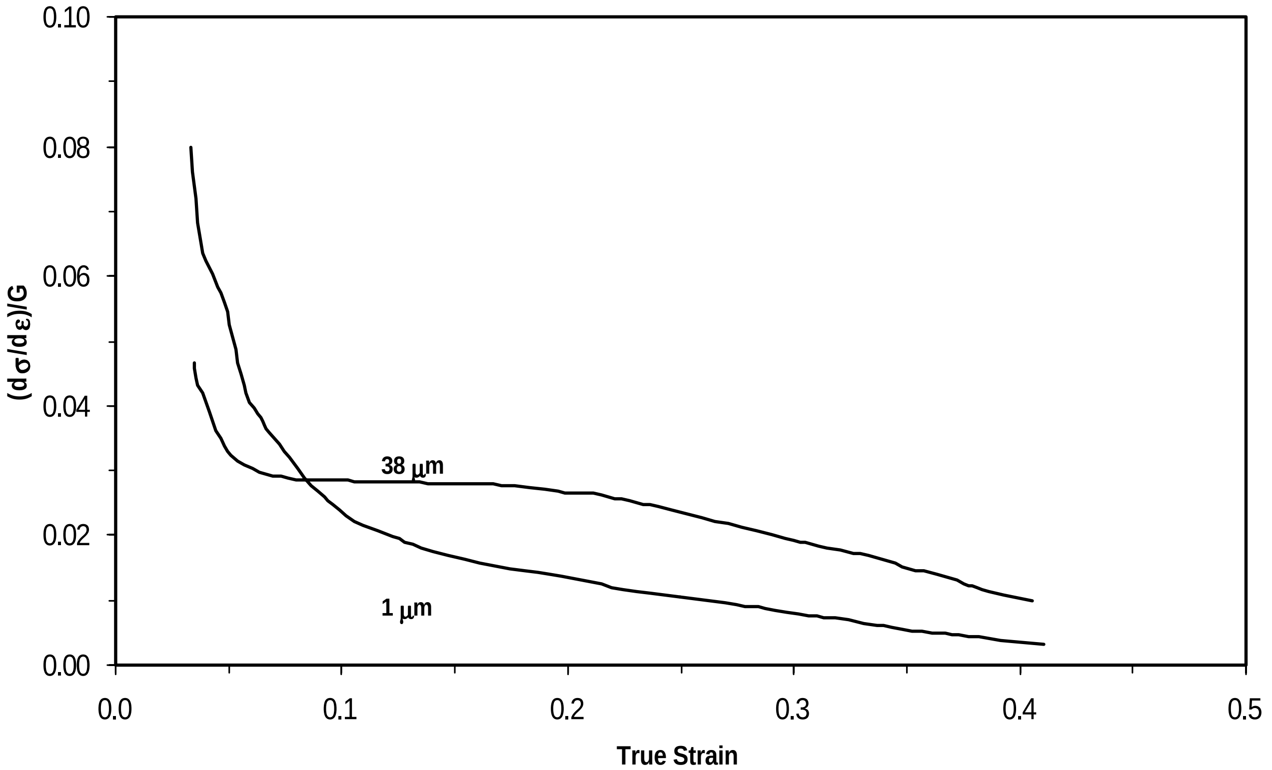
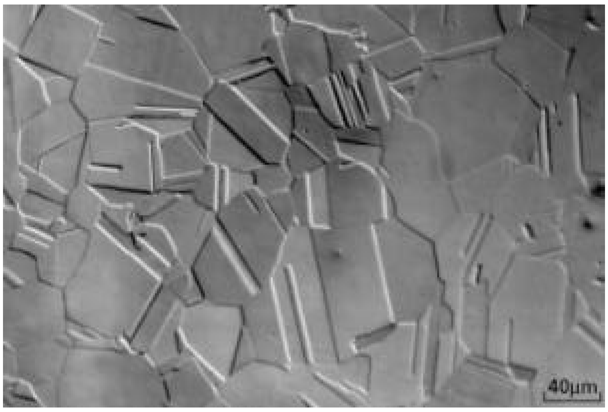
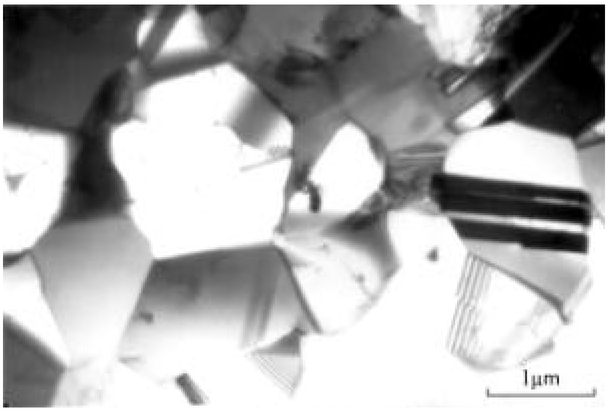



**Figure 2**. Normalized work hardening rate vs. true strain of annealed MP35N alloy with two different grain sizes.




**(a)**




**(b)**

**Figure 3**. (a) Optical micrograph of annealed MP35N alloy with 38 ìm grain size and (b) TEM micrograph of annealed MP35N alloy with 1ìm grain size.

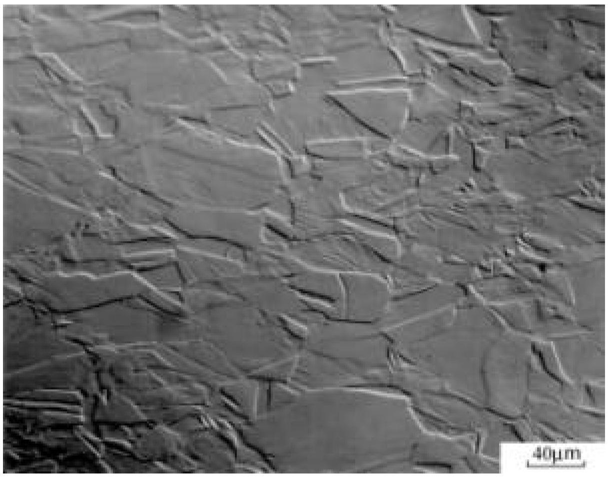
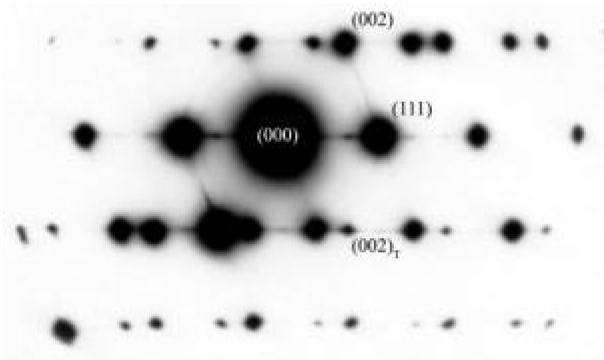



**Figure 3**. Optical micrograph of MP35N alloy with 38 ìm grain size deformed to a strain of 0.18 in tension.


**(a)**




**(b)**

**Figure 5** (a) Bright field (BF) image and (b) diffraction pattern of the sample used in Figure 4.

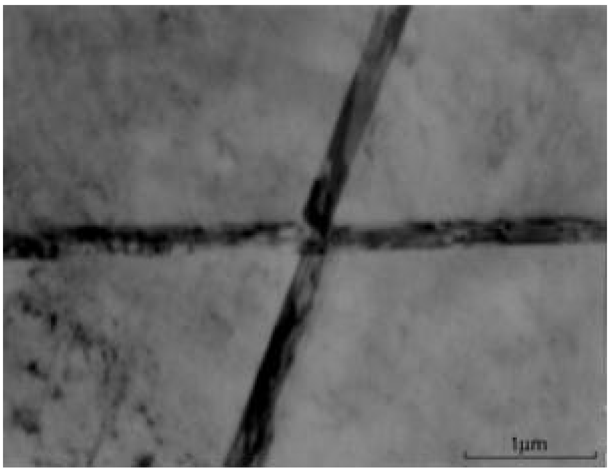



**Figure 6**. TEM micrograph of intersecting twins observed in MP35N alloy with 38 ìm grain size deformed to a strain of 0.4.

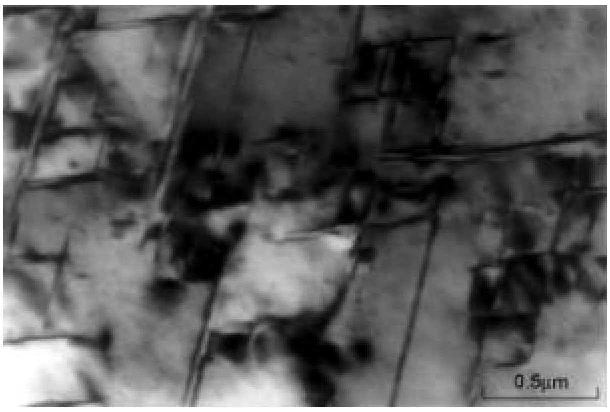



**Figure 7**. TEM micrograph of 1 m grain size MP35N alloy deformed to a strain of 0.32 in tension.

38 μm grain size sample. Between the strain levels of 0.05 and 0.2, a second regime of nearly constant strain hardening rate develops. The third regime with a falling strain hardening rate occurs in the strain range of 0.2-0.4. The hardening rate of the 1 μm grain size alloy, however, does not follow the same trend. In particular, the second regime of constant strain hardening seen in the 38 μm grain size MP35N, is completely absent in the fine grain size alloy. This leads to a lower strain-hardening rate in the fine grain size material at strains larger than about 0.1, compared to that of the coarser sample.

**Optical Microscopy and TEM Observations**
Figures 3(a) and 3(b) show the microstructures of annealed MP35N with 38 and 1 μm grain sizes, respectively. The large number of annealing twins observed in these structures is characteristic of low SFE alloys. Also, the very low dislocation density seen inside the equiaxed grains in Figure 3(b) is a typical feature of recrystallized structures. Figure 4 shows an optical micrograph of a 38 μm grain size alloy deformed to a strain of 0.18 in simple compression. The important feature of this structure is the very large density of deformation markings observed inside the grains. Figure 5 presents the TEM results obtained from the same sample. Figure 5(a) is a bright field (BF) image that shows the plate-like nature of these markings. Figure 5(b) is the [110] diffraction pattern obtained from the same area. Twin spots are clearly seen in this pattern. These results are very similar to those previously reported for MP35N deformed in simple compression [5] and clearly identify the observed markings in Figure 4 as deformation twins. At a strain level of 0.4, two intersecting sets of deformation twins were found in a number of grains. Figure 6 shows an example of intersecting twins formed in this sample. TEM studies on the fine grain size samples deformed to strain levels of 0.05, 0.17, and 0.21 did not show any deformation twins. At a strain level of 0.32, deformation twins were observed in the structure of the 1 μm grain size alloy. Interestingly, these were mostly short twins that formed in some areas within the grains. Only in a few grains long parallel twins were found. Figure 7 shows an example of the short deformation twins observed at this strain level. With increasing plastic strain to 0.42, the density of short deformation twins increased slightly in the fine grain size alloy.

## 4. DISCUSSION

The behavior of the 38 μm grain size MP35N alloy shown in Figures 1 and 2 is in good agreement with that previously reported for low SFE FCC materials of a comparable grain size deformed in simple compression [5,7]. The hardening rate initially decreases with increasing plastic strain (dynamic recovery regime) followed by a second stage of rather constant hardening rate. With further increase in the applied strain, a third regime of decreasing hardening rate is followed. In a tensile test, true stress-true strain and therefore work hardening data are limited by the occurrence of necking and only these three stages of work hardening are usually seen. Previous studies [5,7] using large strain simple compression testing have shown that at strains larger than about 0.7-0.8 a final stage of constant hardening may occur.

While the plastic behavior of the 38 μm grain size sample is in good agreement with that reported in previous studies, the behavior of the fine grain size alloy is unexpected. During the early stages of deformation, the dynamic recovery regime in the fine grain size material occurs with a higher hardening rate, as expected. The second regime of constant hardening rate observed in the 38 μm grain size material, however, is completely missing in the fine grained material. As a result, at strains larger than about 0.1, the work hardening rate of the 1 μm grain size material falls below that of the 38 μm grain size alloy. The microstructural origin of the second regime of hardening in low SFE fcc alloys has been recently investigated in simple compression testing. It has been reported [5,7] that the onset of this uniform hardening regime in low SFE materials in simple compression coincides with the formation of extensive primary twins in the microstructure. In the present study, formation of deformation twins in a sample deformed to a strain of 0.18 in tension is also confirmed (Figure 5). TEM studies on the fine grain size material, however, did not show any evidence of twin formation up to a strain of 0.32. Short deformation twins found at this strain level in the fine grain size material are probably a result of the lattice misorientations within the grains caused by inhomogeneous plastic deformation. Formation of these twins has no significant impact on the hardening behavior of the material, as seen from Figure 2. This may be explained considering local formation of twins which greatly reduces their effectiveness in hindering slip. Also, due to the higher stress level compared to the large grained material, dynamic recovery process is more efficient at similar strain levels. As a result of these effects, hardening behavior of the fine grained material shows a continuous drop with strain, a typical behavior of high SFE materials during Stage III of work hardening. These results clearly demonstrate the role of deformation twins in enhancing the work hardening rate of low SFE FCC alloys. Also, the need to overcome a critical twinning stress for formation of deformation twins is implied.

These observations may not be explained based on the previous models for the effect of grain size on strain hardening behavior of FCC polycrystals [11-15]. Studies on grain size effects up to moderate strains (in tension) have shown that the effect of grain size on *flow* stress of materials such as α-brass can be expressed by a Hall-Petch type equation while such a relationship can not describe the grain size effects in copper and aluminum [13,15]. To rationalize the grain size effects in these materials, a composite model for deformation of polycrystals has been proposed by Kocks [11] and is further developed by Thompson and coworkers [13]. The main idea of the composite model is that the regions near grain boundaries harden differently from those at the center of the grains. Using the Ashby's idea of "statistically stored" and "geometrically necessary" dislocations, a flow stress relation for each region of a grain is used. Away from the grain boundaries, the dislocation density is of statistically stored type that behaves similarly to that created during deformation of single crystals. In the grain boundary region, however, geometrically necessary dislocations dominate the dislocation population. These are generated to maintain the continuity of the polycrystals and, at small strains, are grain size dependent. As the strain increases, the statistical term becomes dominant and thus at large strains the grain size dependence of flow stress becomes negligible. The importance of statistical and geometrical terms has been also related to the slip character of the material. It is suggested that the behavior of high SFE materials such as aluminum is mainly controlled by statistical term while the geometrical term plays the dominant role in the behavior of low SFE alloys such as α-brass up to large strains.

The models mentioned above are solely based on grain size effects on the dislocation structure. These models do not include the effect of grain size on the microstructural features other than dislocation structures that may contribute to work hardening response of a material. It is now well established, however, that deformation twinning plays an important role in strain hardening behavior of low SFE FCC materials [5,16,17]. Also, it has been reported that twin formation becomes more difficult with decreasing the grain size and may be completely inhibited in very fine grain size materials [18]. It is, therefore, possible that a low SFE polycrystal with a very small grain size exhibit a decreased rate of work hardening compared to a coarse grained material. This is consistent with the data presented in Figures 1 and 2, which show a lower strain-hardening rate for the fine grain size alloy at intermediate to large strain levels. Strikingly, in the strain range of 0.09-0.2, where 38 μm grain size MP35N shows an enhanced hardening rate due to extensive twinning, TEM observations on the fine grain size alloy clearly show the absence of extensive twinning. These observations provide a further support for the idea that deformation twins may effectively hinder dislocation slip and significantly contribute to the hardening rate of low SFE materials. It is therefore necessary to modify the current plasticity models for FCC polycrystals to include deformation twinning as a second deformation mechanism. It is also interesting to study the plastic behavior of the 1 μm grain size MP35N in simple compression testing and to characterize the hardening behavior of the material at large strains. This is currently under investigation.

## 5. CONCLUSIONS

**1.** Strain hardening behavior of MP35N alloy with 1μm grain size in uniaxial tension is distinctly different from that of 38 μm grain size alloy. The second stage of hardening observed in the 38 μm grain size MP35N in the strain range of 0.09 to 0.2, is completely absent in the fine grain size alloy.

**2.** TEM studies show that the absence of the second hardening regime in the fine grain size MP35N alloy coincides with the absence of deformation twins in the microstructure. This implies a need to overcome a critical twinning stress for deformation twinning to occur.

3. At a strain of 0.32, short deformation twins were observed in the fine grain size material. It is suggested that formation of these twins is a consequence of misorientations developed within the grains as a result of inhomogeneous plastic deformation.

4. Results presented in this study clearly show the need to include deformation twinning in the plasticity models for low SFE FCC polycrystals. The current dislocation-based models are incapable of predicting the observed hardening behavior of these materials.

## 6. ACKNOWLEDGMENTS

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