Effect of Cold Deformation on Microstructure and Mechanical Behavior of GH3535 Alloy Postprint

Authors: Fan Jinhui, Chen Kexin, Liang Jianping, Li Zhijun, Li Xiaoke

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Abstract

Tensile tests with various deformation amounts were conducted on GH3535 alloy to investigate the effect of cold deformation on its mechanical properties. The microstructures after cold deformation were characterized by OM and TEM, and the deformation mechanisms of work hardening in GH3535 alloy were analyzed in conjunction with true stress-true strain curves. The results demonstrate that GH3535 alloy exhibits significant work hardening characteristics: cold deformation enhances strength and hardness but reduces ductility. With increasing deformation amount, grains elongate along the deformation direction, and twins become more abundant and coarser. The work hardening behavior of GH3535 alloy follows the Ludwigson model. As the cold deformation amount increases, the work hardening exponent decreases, and the deformation mechanism gradually transitions from single slip and twinning to cross-slip and twinning. For deformation amounts below 30%, work hardening is primarily attributed to the long-range stress fields of dislocations and twins, whereas for deformation amounts exceeding 30%, it is mainly caused by the short-range stress fields of dislocations and deformation twins.

Full Text

Effect of Cold Deformation on Microstructure and Mechanical Behavior of Ni-based High Temperature Alloy GH3535

FAN Jinhui[1], **CHEN Kexin**[1,2,3], **LIANG Jianping**[2,3], **LI Zhijun**[2,3], **LI Xiaoke**[2,3]

[1] University of Chinese Academy of Sciences, Beijing 100049, China
[2] Shanghai Institute of Applied Physics, Chinese Academy of Sciences, Shanghai 201800, China

[3] Key Laboratory of Nuclear Radiation and Nuclear Energy Technology, Chinese Academy of Sciences, Shanghai 201800, China

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Abstract

The effects of extensometer-induced cold-tensile deformation on the microstructure and mechanical properties of Ni-based high temperature alloy GH3535 were investigated using optical microscopy (OM), transmission electron microscopy (TEM), and measurement of true stress-true strain curves. The results show that GH3535 alloy exhibits strong work hardening characteristics; cold deformation significantly increases its strength and hardness while decreasing ductility. With increasing deformation degree, grains become elongated along the deformation direction and twins become more numerous and coarser. The work hardening kinetics of GH3535 alloy follow the Ludwigson model. The work hardening exponent decreases with increasing cold deformation, and the deformation mechanism gradually transitions from single slip and twinning to cross slip and twinning. When the deformation degree is below 30%, work hardening is mainly caused by the dislocation long-range stress field and twins; when the deformation degree exceeds 30%, it is primarily caused by the dislocation short-range stress field and deformation twins.

Keywords: metallic materials, nickel-based superalloy, cold deformation, microstructure, mechanical property

Introduction

Molten salt reactors have attracted significant attention in the international nuclear energy community due to their inherent advantages, including better utilization of thorium resources, excellent safety characteristics, and high breeding ratios. They represent an ideal reactor type for implementing the thorium-uranium fuel cycle to achieve ^{233}U breeding and have been listed as one of six candidate Generation IV advanced nuclear reactor systems, being the only liquid-fuel reactor among them. The structural materials used in molten salt reactors should possess high-temperature resistance, corrosion resistance, and neutron irradiation resistance. Hastelloy N alloy, a Ni-Mo-Cr corrosion-resistant alloy specifically developed for molten salt reactor environments, features a solid solution-strengthened face-centered cubic structure. Its high molybdenum and low chromium content provide excellent performance in high-temperature molten salt environments, making it suitable for manufacturing structural components such as loop piping and vessels for molten salt reactors.

Current research on Hastelloy N alloy has primarily focused on irradiation, corrosion, and precipitate behavior. While Oak Ridge National Laboratory studied the effects of cold deformation on the mechanical properties of Hastelloy N alloy, they did not systematically analyze the work hardening deformation mechanisms in conjunction with true stress-true strain curves and microstructural features at different deformation stages. This study investigates the effects of various deformation degrees on the microstructure and mechanical behavior of GH3535 alloy, analyzes the true stress-true strain response curves using the Ludwigson model, and examines the work hardening deformation mechanisms.

Experimental Methods

The chemical composition of the experimental GH3535 alloy bar (in wt%) was: C 0.013, Cr 7.08, Mn 0.67, Mo 16.7, Fe 4.2, Si 0.45, Co 0.009, Cu 0.006, P 0.005, Ti 0.004, S 0.0005, B 0.001, with Ni balance. The material was first subjected to solution treatment at 1177°C for 40 minutes, then machined into standard round tensile specimens with a gauge length of 25 mm.

Tensile tests were conducted on a Zwick/Roell Z100 universal testing machine. Using a standard extensometer, specimens were stretched to different pre-deformation levels (4%, 7%, 10%, 20%, 30%, 40%) and then unloaded to prepare samples with various deformation amounts. According to GB/T228.1-2010 "Metallic Materials—Tensile Testing at Ambient Temperature," the gauge length of these pre-deformed specimens was re-calibrated using an extensometer, and tensile tests were performed until fracture to compare their mechanical properties.

Metallographic specimens were prepared along the tensile direction and observed using an Axio Imager M2m optical microscope. Hardness (HV) was measured on a Zwick/Roell ZHV30 Vickers hardness tester. Transmission electron microscopy (TEM) foil specimens were taken from the cross-section of the uniformly deformed portion of tensile samples. The required samples were cut into 0.5 mm thick slices using wire electrical discharge machining, ground to 100 m using 1500-grit sandpaper, punched into 3 mm diameter discs, and then thinned using a TenuPol-5 twin-jet electropolishing instrument. The electrolyte consisted of 5% $HClO_4$ + 95% C_2H_5OH solution (temperature: -20 to -30°C, voltage: 30-50 V). After electropolishing, specimens were immediately cleaned with alcohol, dried, and examined using an FEI Tecnai G2 transmission electron microscope.

2.1 Microstructure After Cold Deformation

[Figure 1: see original paper] shows the microstructures of GH3535 alloy at different deformation levels. As shown in Figure 1a, the solution-treated material exhibits a typical austenitic structure with equiaxed grains averaging 35 m in size, containing numerous annealing twins with clearly visible continuous and straight twin boundaries. Figures 1b-g reveal that with increasing deformation degree, the degree of grain deformation progressively increases, grains gradually elongate along the tensile direction, and the number of deformation twins increases with twins becoming coarser. Additionally, at 20% deformation, some twin boundaries within grains begin to bend while others remain continuous and straight, indicating that the fully coherent relationship of twin boundaries starts to be disrupted, meaning twins begin to impede deformation. When the deformation degree increases to 30% and 40%, most twin boundaries within grains are observed to have bent, indicating further disruption of the fully coherent twin boundary relationship.

2.2 Room-Temperature Mechanical Properties After Cold Deformation

The relationship between cold deformation degree and true stress-true strain curves for GH3535 alloy is shown in [Figure 2: see original paper]. It can be seen that material strength increases with deformation degree, while the true strain corresponding to the maximum tensile true stress shows a decreasing trend, demonstrating significant work hardening behavior. [Figure 3: see original paper] shows that both yield strength and ultimate tensile strength gradually increase with deformation degree, while plasticity decreases. Additionally, the difference between yield strength and ultimate tensile strength becomes smaller with increasing deformation, indicating that the yield ratio increases and formability gradually decreases. At 40% deformation, yield strength and ultimate tensile strength become equal. This occurs because yield strength is primarily controlled by large-scale dislocation slip; cold deformation generates dislocations within the material, and greater deformation leads to higher dislocation density and stronger dislocation interactions, thus significantly increasing yield strength. In contrast, ultimate tensile strength is mainly controlled by microcrack initiation and propagation, with limited influence from dislocation motion, so cold deformation has a relatively smaller effect on increasing ultimate tensile strength. Dislocation interactions reduce dislocation mobility, increase flow stress, and the strengthening effect of cell structures formed by high dislocation density becomes pronounced, thereby decreasing the work hardening rate. The combined effect of these factors leads to a significant reduction in uniform plasticity.

[Figure 4: see original paper] presents the hardness curves of different materials after various cold deformation degrees. The hardness of undeformed GH3535 alloy is HV 193, which increases significantly with deformation degree, reaching HV 390 at 40% deformation, demonstrating extremely strong cold work hardening capability. The figure also shows that the cold work hardening capacity of GH3535 alloy is intermediate between Hastelloy C276 and Hastelloy C22 alloys.

2.3 Discussion

The steady-state flow region of true stress-true strain curves for austenitic steels shows a concave-upward shape when plotted on a logarithmic scale, making them unsuitable for description using the traditional Hollomon equation ($\sigma =$ K ^{n}, where σ is true stress,   is true strain, K is the strength coefficient, and n is the work hardening exponent). To address this, Swift and others proposed several models for fitting the steady-state flow curves of austenitic steels, among which Ludwigson' s model has received widespread attention for describing true stress-true strain curves of FCC metals and alloys:

$$\sigma = \mathrm{K}_1\ ^{n1} + \exp(\mathrm{K}_2 + \mathrm{n}_2\ )$$

where K_1 and n_1 correspond to K and n in the Hollomon equation; K_2 represents the logarithmic deviation of the Hollomon equation ($\sigma =$ K ^{n}) at low strains, and n_2 represents the rate at which the ratio of short-range to long-range forces decreases with increasing plastic strain. The Ludwigson model also defines a critical strain _L, which indicates that when true strain exceeds this value, the material' s true stress-true strain curve can be described by the Hollomon model. This parameter serves as a measure of the transition in deformation mechanism from single slip to cross slip.

[Figure 5: see original paper] shows the log-log scale true stress-true strain curves for GH3535 alloy at different deformation degrees. The curves exhibit a clear concave-upward shape that becomes less pronounced and approaches linearity with increasing deformation degree. The high-strain regions of all curves tend toward linearity with decreasing slope. At 30% deformation, the curve is essentially linear, indicating that the traditional Hollomon equation can be applied.

Regression analysis of the steady-state flow stage of GH3535 alloy' s true stress-true strain curves using the Ludwigson model yields the results shown in . The Ludwigson model provides an excellent description of the curves at all deformation levels. In this model, n_1 reflects a material' s resistance to continued plastic deformation; larger n_1 values indicate more severe work hardening during forming. The n_1 value is related to the material' s stacking fault energy —low stacking fault energy corresponds to high n_1 values, while high stacking fault energy corresponds to low n_1 values. Since stacking fault energy reflects the ease of cross slip, lower stacking fault energy makes cross slip more difficult. Therefore, the deformation mechanism can be analyzed from n_1 values.

As shown in , the work hardening exponent n_1 decreases rapidly with increasing deformation degree, indicating that cold deformation increases the stacking fault energy of GH3535 alloy. When the deformation degree reaches 30%, the critical strain _L becomes negative, suggesting that the material' s deformation mechanism exhibits cross slip from the initial deformation stage. Therefore, the Ludwigson model can be used for regression analysis of GH3535 alloy' s true stress-true strain curves at deformation levels below this threshold, where the deformation mechanism is primarily single-plane slip. Above this deformation level, cross slip dominates and the Hollomon equation can be used for regression analysis.

The table also shows that when the deformation degree is below 30%, the absolute value of n_2 increases with deformation, indicating that the dislocation long-range stress field increases while short-range stress field effects decrease. In this regime, work hardening is mainly caused by the dislocation long-range stress field, corresponding to increasing numbers of grains undergoing dislocation motion and increasing dislocation density, though short-range stress fields from dislocation interactions are not yet significant. At 30% deformation, n_2 becomes positive, and the short-range stress field effect increases rapidly and becomes dominant. At this stage, work hardening in GH3535 alloy primarily originates from the dislocation short-range stress field, with activation of multiple slip systems, dislocation mutual intersection, and increased slip difficulty. Additionally, twins in the microstructure strongly impede slip, increasing deformation resistance. With further deformation, massive dislocation multiplication creates even stronger short-range stress fields, further increasing deformation resistance.

The cold-deformed microstructures of GH3535 alloy show that twin density and size increase with deformation degree, demonstrating that twinning is an important strengthening mechanism during cold deformation. Mahajan and others proposed that deformation twins nucleate when slip is impeded, through the movement of partial dislocations at stress concentration sites, and that twin quantity and thickness significantly affect material properties. Deformation twin formation and growth are closely related to cross slip. Lower stacking fault energy makes cross slip more difficult, reducing dislocation cross-slip activity and restricting twinning partial dislocation motion on continuous {111} planes, thereby inhibiting twin growth. Additionally, matrix dislocations interact with twins, preventing twin growth. This requires sufficiently high stress concentration at twin nucleation sites to overcome dislocation repulsion, with this stress concentration increasing with stacking fault energy—indicating that low stacking fault energy makes deformation twin growth difficult. As the work hardening exponent n_1 decreases with increasing deformation degree, the alloy’ s stacking fault energy increases, making cross slip easier and creating more severe stress concentration at twin nucleation sites. Particularly after 30% deformation where cross slip dominates, twin density increases and deformation twins become coarser, resulting in more pronounced work hardening. This occurs because deformation twin formation effectively refines grains and increases flow stress, while twins create additional obstacles for dislocation motion, further increasing flow stress.

TEM observations of GH3535 alloy at different deformation levels reveal deformation characteristics similar to those reported by Wang. The process can be divided into three stages: (1) twinning and slip co-participation, (2) twinning impeding slip, and (3) slip cutting through twins. As shown in [Figure 6: see original paper], at 0-20% deformation, dislocations undergo planar slip, dislocations from different slip systems mutually intersect and pin each other, leading to rapid dislocation multiplication and formation of dense pile-up groups. Twin density increases (Figures 6a,b), resulting in effective grain size refinement. In this stage, twins and slip co-participate in deformation, and the formation of dislocation pile-ups combined with increasing twin density causes the work hardening exponent n_1 to decrease and strength to increase. With further deformation, more dislocation pile-up groups and deformation twins form (Figure 6c), twins strongly impede dislocation slip motion (Figure 6d), and some twins undergo local bending deformation with damaged twin boundaries. This indicates that twin boundary obstruction is insufficient to resist stress concentration from dislocation pile-ups, while massive dislocation pile-ups cause extended dislocations to constrict and participate in deformation through cross slip. In this stage, twins impede slip, and the slip mode transitions from single slip to cross slip, causing the work hardening exponent n_1 to decrease further and strength to increase further, corresponding to the 20%-30% deformation range. At even higher deformation levels, massive dislocation multiplication (Figure 6e) severely damages twin coherency, and dislocation slip begins to cut through twins, reducing their impeding effect.

In summary, work hardening in GH3535 alloy during cold deformation results primarily from the combined effects of dislocation strengthening and twin strengthening. When the deformation degree is below 30%, work hardening is mainly caused by the dislocation long-range stress field and twins; when the deformation degree exceeds 30%, it is primarily caused by the dislocation short-range stress field and deformation twins.

Conclusions

1. With increasing cold deformation degree, the deformation degree of GH3535 alloy grains progressively increases, grains gradually elongate along the tensile direction, and twins become more numerous and coarser.
2. GH3535 alloy exhibits significant work hardening characteristics. Cold deformation substantially increases strength and hardness while decreasing plasticity. Its cold work hardening capacity is intermediate between Hastelloy C276 and Hastelloy C22 alloys.
3. When the cold deformation degree is below 30%, the log-log true stress-true strain curves of GH3535 alloy show a concave-upward shape that can be analyzed using the Ludwigson model. Above this deformation level, the curves approach linearity and can also be analyzed using the traditional Hollomon equation.
4. The Ludwigson model effectively describes the true stress-true strain curves of GH3535 alloy, with model parameters that well explain the material' s deformation behavior. The work hardening exponent of GH3535 alloy decreases with increasing cold deformation degree, and the deformation mechanism transitions from single slip and twinning to cross slip and twinning.

5. Work hardening generated during cold deformation of GH3535 alloy results primarily from the combined effects of dislocation strengthening and twin strengthening. When the deformation degree is below 30%, work hardening is mainly caused by the dislocation long-range stress field and twins; when the deformation degree exceeds 30%, it is primarily caused by the dislocation short-range stress field and deformation twins.

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Note: Figure translations are in progress. See original paper for figures.