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A COMPARATIVE ANALYSIS ON VARIOUS STRATEGIES TO PRODUCE NANO-CRYSTALLINE AUSTENITIC STAINLESS STEEL

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A Comparative Analysis on Various Strategies to Produce Nano-Crystalline Austenitic Stainless Steel

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Abstract – A Super304H austenitic stainless steel, Fe - 0.1 %C - 0.12%N - 0.1 %Si - 0.95%Mn - 18.4%Cr - 7.85%Ni - 3.2%Cu - 0.5%Nb - 0.01%P - 0.006%S (all in mass%), with an average grain size of about 10 μm was used as the starting material. The multiple forging was carried out by means of multi-pass compressions at room temperature. The hardness of 6 GPa and the yield strength of 1430 MPa were achieved after total strain of 4. The strengthening during the multiple forging resulted from the formation of almost equiaxed nanocrystalline structure with an average grain size of about 30 nm. The softening behaviour of the nanocrystalline samples was studied by means of isochronal annealing at temperatures of 500 to 700 °C for 30 min. The structural mechanisms responsible for the grain refinement during the large strain cold working and those operating upon the subsequent annealing and their effect on the mechanical properties are considered.

The development of nanocrystalline structures in austenitic stainless steels during large strain cold rolling and their tensile behavior were studied. The cold rolling to total equivalent strains above 2 was accompanied by the evolution of nanocrystalline structures with the transverse grain size of about 100 nm. The development of deformation twinning and martensitic transformation during cold working promoted the fast kinetics of structural changes. The development of nanocrystalline structures resulted in significant strengthening.

More than fourfold increase in the yield strength was achieved. The strengthening of nanocrystalline steels after severe plastic deformation was considered as a concurrent operation of two strengthening mechanisms, which were attributed to grain size and internal stress. The contribution of internal stresses to the yield strength is comparable with that from grain size strengthening.

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INTRODUCTION

The development of ultrafine grained or nanocrystalline structures is of the utmost importance for austenitic stainless steels. These materials are characterized by relatively low yield strength after ordinary thermomechanical processing . The structural strengthening by means of large strain deformations has been shown as an advanced approach for production of high strength austenitic stainless steels. The aim of the present study is to clarify the mechanisms of structural changes leading to the development of nanocrystalline structures in austenitic stainless steels during cold rolling and to elucidate the relationship between the developed nanocrystalline structures and the mechanical properties. Two types of steels, i.e. 316L and 304L, are used as typical representatives of frequently used austenitic stainless steels.

Austenitic stainless steels (ASSs) have many advantages from a metallurgical point of view. They can be made soft enough with yield strength about 200 MPa, but they can also be made incredibly strong by cold work up to yield strengths of over 2,000 MPa . Alloy AISI 321 is a titanium-stabilized stainless steel, which offered an excellent resistance to intergranular corrosion. It is known that grain size is a key microstructural factor affecting physical and mechanical properties of materials . Grain refinement can increase the strength as it improves toughness of the metals . Therefore, grain refinement has been attracting considerable leaning from material engineering science. In recent years, there has been an interest in developing nano/ultrafine grain stainless steels to get high strength/good ductility alloys. For this purpose, several techniques including advanced thermo-mechanical treatment (ATP) and severe plastic deformation (SPD) techniques like high pressure torsion (HPT), equal channel angular pressing (ECAP) and accumulative roll bonding (ARB) have been used. Wang et al. created nano/ultrafine grains in AISI 316 steel using HPT technique at room temperature followed by annealing at 500 _C for 1 h.

They attained yield strength about 2,230 MPa. Qu et al. obtained bulk nanocrystalline grain structure in AISI 304L stainless steel by means of ECAP at room temperature.

They acclaimed that two types of nanostructures were formed: nanocrystalline deformation-induced martensite

(DIM) with a mean grain size of 74 nm and nanocrystalline austenite with a size of 31 nm characterized by high density of deformation twins. Reports have indicated that ASSs undergo martensitic transformation during SPD at room temperature. Martensitic transformation leads to an additional grain refinement and a dual phase austenitic-martensitic nanocrystalline structure.

Stainless steels are very important materials in regard of economic and technological point of view. To date, there are more than 200 different types of alloys that belong to stainless steel family. Stainless steels are used in a wide variety of services in which primary considerations are long service life, reliability, appearance and sanitary factors. Important interest is the use of these materials in the medical field. Presently, eg. most biomedical implants consist of a stainless steel framework. However, widely used 316L steel is not fully biocompatible, and has induced high occurrences of restenosis and thrombosis. Moreover, nickel ions produced due to corrosion are reported to cause allergies and even cancer. Nickel free austenitic stainless steels with nanostructure are one of the promising materials that fulfill these challenging criteria.

DEVELOPMENT OF NANOCRYSTALLINE 304L STAINLESS STEEL BY LARGE STRAIN COLD WORKING

The large strain deformations are considered as promising methods for development of advanced structural steels and alloys with enhanced mechanical properties. The significant improvement of mechanical properties of metallic materials subjected to severe plastic deformations is commonly attributed to the strain-induced ultrafine-grained or, even, nanocrystalline structures. The ultrafine-grained materials have been shown to possess a unique combination of high strength and surprisingly large ductility . The efficiency of cold working for processing the high-strength ultrafine-grained/nanocrystalline products depends remarkably on the kinetics of grain refinement during plastic deformation. Austenitic stainless steels are typical representative of metallic materials exhibiting rapid grain refinement upon cold working. The grain refinement in these steels is promoted by an intensive grain subdivision, which is associated with deformation twinning followed by strain-induced martensitic transformation. Therefore, the austenitic stainless steels can be easily produced in high-strength ultrafine-grained/nanocrystalline state by conventional cold working technique like plate rolling . In spite of a number of research works dealing with nanocrystalline stainless steels processed by large strain cold working, however, the mechanisms of microstructure evolution, *i.e.*, a role of deformation twinning and strain-induced martensite, and their contribution to strengthening are still unclear.

The strengthening of metallic materials processed by large strain deformation is generally discussed in terms of either grain boundary strengthening or dislocation strengthening. The former is commonly evaluated as $\sigma_{GB} = K_\varepsilon D^{-0.5}$, where D is the grain size and K_ε is a constant; and the latter is related to a square root of dislocation density as $\sigma_{DISL} = \alpha G b \rho^{0.5}$, where α, G, and b are a constant, the shear modulus, and the Burgers vector, respectively. Assuming that the grain boundary strengthening and the dislocation strengthening contribute independently to overall strength, a modified Hall-Petch-type relationship has been recently introduced to relate the yield strength of ultrafine-grainediianociystalline materials processed by severe plastic deformation to their microstructural parameters, *i.e.,* the grain size and dislocation density, in the following form:

Here, σ_0 is the strength of dislocation-free single crystal. Recent studies 011 severely deformed quasi-single phase ultrafine-grained/nanocrystalline materials have shown that the contribution from dislocation strengthening exceeds remarkably that from grain boundaries. However, the strengthening mechanisms for ultrafine-grained/nanociystalline materials such as metastable austenitic stainless steels, which experience martensitic phase transformation during cold working, have not been studied.

EXPERIMENTAL PROCEDURE

A 316L-type austenitic stainless steel (Fe-0.04%C-0.4%Si-1.7%Mn-17.3%Cr-10.7%Ni-2%Mo- 0.04%P-0.05%S) and a 304L-type austenitic stainless steel (Fe-0.05%C-0.4%Si-1.7%Mn-18.2%Cr- 8.8%Ni-0.05%P-0.04%S) were usecl as starting material. The steel samples were hot rolled and then annealed at 1100°C for 10 min. The initial annealed grain sizes were 21 and 24 μm in the 316L and 304L steel samples, respectively. The cold working was carried out by caliber rolling 9.2 nun x 9.2 nun square bars into 1.25 mm x 1.25 nun square bars at ambient temperature, leading to a total equivalent strain of 4.

Structural investigations were performed close to the sample axis on sections parallel to the rolling axis, using a Nova Nanosem 450 scanning electron microscope (SEM) incorporating an orientation imaging microscopy (OIM) system and a JEM-2100 transmission electron microscope (TEM). The grain sizes were measured on perpendicular to the rolling axis by a linear intercept method, including all boundaries with disorientation $\theta \geq 15°$ revealed by OIM micrographs. The volume fractions of the austenite were averaged through OIM, X-ray analysis and magnetic induction method. The hardness of rolled samples was studied using Vickers hardness tests with a load of 3N with a corresponding indent diagonal of about 35 urn. The tensile tests were carried out by using specimens with a gage length of $L_0 = 5.65\sqrt{S_0}$, where S_0 is the cross-sectional area.

CONCLUSION

The development of nanocrystalline structures in 316L and 304L austenitic stainless steels during large strain cold rolling was studied. The present steels are characterized by fast kinetics of grain refinement. The cold rolling to total strains above 2 resulted in the development of nanocrystalline structures with the transverse grain sizes of about 100 nm. The rapid grain refinement was promoted by the development of deformation twinning and martensitic transformation. The martensitic transformation developed more readily in the 304L steel samples, leading to somewhat finer average austenite/ferrite grains as compared to 316L ones. The development of nanocrystalline structures resulted in significant strengthening, which was considered as a result of the grain size strengthening (Hall-Petch relationship) and the internal stresses, which were attributed to the large internal distortions involved by severe deformation.

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