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Elucidating microstructural evolution and strengthening mechanisms in nanocrystalline surface induced by surface mechanical attrition treatment of stainless steel

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ABSTRACT

Surface mechanical attrition treatment (SMAT) is a high strain and strain rate severe plastic deformation (SPD) technique for surface nanocrystallization of metals. The aim of this study was to investigate the mechanism of nanocrystallization and strengthening in a medium stacking fault energy 316 L austenitic stainless steel during SMAT. The paramount role of microband and shear band formation in nanocrystallization is outlined, as opposed to deformation twinning previously reported in low SFE austenitic stainless steels. Shear bands undergo dynamic recrystallization and recrystallization twinning to produce ultra-fine grains in contrast to twin-twin intersections in low SFE stainless steel. The ultra-fine grains further sub-divide into smaller cells with initially low misorientation. Nanocrystallization occurs when misorientation between these cells increases with further strain. The additivity of strengthening by dislocation density and grain size is studied. Dislocation density was neglected in previous studies while studying strengthening mechanisms in SMAT processed materials. This study illustrates that dislocation density cannot be ignored as the strengthening mechanism in SMAT process. The grain size and dislocation density both significantly contribute to overall strengthening in SMAT processed microstructure.

1. Introduction

Surface mechanical attrition treatment (SMAT) is a severe plastic deformation (SPD) process that induces nanocrystallization at the surface [1]. SMAT is a process that is derived from the conventional shot peening process. The shot peening based processes are frequently used to engender surface nanocrystallization. Nanocrystallization can also be achieved using conventional shot peening using severe parameters and the process is referred to as severe shot peening [2,3]. The fundamental difference between the conventional shot peening and SMAT is that the impact of balls is in random directions in SMAT compared to the well-defined impact angles in shot peening. High strain and strain rates combined with random impact directions facilitate the process of nanocrystallization. However, multiple impact directions are also possible with conventional shot peening by varying the incidence angle of peening media with respect to the processed specimen [4]. Shot peening based processes like SMAT and severe shot peening as a surface modification technique have shown promise in improving fatigue/corrosion-fatigue strength, wear resistance, corrosion resistance and bioactivity of metals [5–10].

The mechanism of nanocrystallization at the surface during SMAT has been studied extensively for certain metals and alloys. Nanocrystallization in pure Fe has been attributed to the formation of dense dislocation walls/dislocation tangles (DDWs/DTs). DDW/DT acquire higher misorientations and transform into subgrain boundaries [11]. The subgrains further divide by a similar mechanism and finally attain a stable grain size in nanometer scale. Zhang et al. [12] carried out a comprehensive study on the nanocrystallization mechanism in 304 stainless steel (SS) and reported the extensive formation of intersecting ultra-fine twins that lead to grain sub-division. Emergence of randomly oriented grains was reported and it has been speculated that they possibly result from grain rotation, grain boundary sliding or recrystallization via nucleation. However, no clear evidence was provided for the proposed mechanism. Strain induced martensitic transformation is also reported to facilitate the nanocrystallization by providing high angle grain boundaries. Zhu et al. [13] studied the mechanism of

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List of abbreviations and symbols:

Bs type	Brass type shear band
CB	Cell block
CDRX	Continuous dynamic recrystallization
CMWP	Convoluted multiple whole profile
Cu-type	Copper type shear band
DDW	Dense dislocation wall
DRX	Dynamic recrystallization
DT	Dislocation tangle
HP	Hall-Petch
IQ	Image quality
IPF	Inverse pole figure
SB	Shear band
SFE	Stacking fault energy
SMAT	Surface mechanical attrition treatment
SPD	Severe plastic deformation
SS	Stainless steel
$< x>_{area}$	area average crystallite size
ρ	Dislocation density
M	Dislocation arrangement parameter
q	Strain anisotropy factor
β	Twin boundary frequency
d_{twin}	Average distance between twin boundaries
σ	Yield stress
σ_0	friction stress
M_T	Taylor factor
G	Shear modulus
α	Geometrical factor in Taylor hardening
K	Hall-Petch constant
D_X	Grain size measured by CMWP ($= < x>_{area}$)
H_v	Micro-Vickers hardness

nanocrystallization in pure Ti. Initial grain sub-division was ascribed to twin-twin intersections, DDWs and microbands leading to low angle boundaries. The subsequent evolution of the nanocrystalline grains was suggested to occur by rotation recrystallization or in other words continuous dynamic recrystallization (CDRX) [14].

Indeed the mechanism of nanocrystallization involving processes like deformation and recrystallization vary based on the material properties. An important material parameter that affects deformation mechanism is the stacking fault energy (SFE) of the material. For example, recently it has been reported [15,16] that for intermediate SFE range Ni-40Co material, the deformation was dominated by slip up to a strain of 70% followed by shear banding. However, for low SFE Ni-60Co slip was active only during initial deformation level, twinning for intermediate deformation level and shear banding for higher deformation level. The SFE of austenitic stainless steels (SS) is reported to be dependent on the composition. For example, the addition of Al to high Mn containing austenitic SS increases its SFE. Hwang et al. [17] studied the effect of SFE on high strain rate deformation in these alloys. The microstructure was found to be dominated by twins and martensite in low SFE SS whereas dislocation tangles and cells were found in the highest SFE SS. Lower SFE in these SS has been shown to promote formation of martensite and shear bands (SBs) [18].

The nanocrystallization during SMAT of 316L SS has not been studied in detail [5,19]. The mechanism of nanocrystallization in 316L SS has been proposed it to be similar to that in 304 SS. However, based on the above observation it is likely that mechanism of nanocrystallization in 316L SS will be different from 304 SS due to the difference in their SFE. 304 SS is a low SFE alloy (18 mJ m^2) whereas 316L is a medium SFE alloy (64 mJ/m^2) [20]. Analogous to the investigation on Ni-Co alloys, it is likely that the deformation mechanisms in the two alloys will be different.

Another important aspect associated with SMAT is the strengthening mechanism. The major source of strengthening in SMAT processed 316 L SS has been attributed to Hall-Petch (HP) effect [5,21]. However, these studies did not estimate dislocation density and therefore neglected its effect on strengthening. HP relationship was first proposed for annealed steels with a grain size greater than 1 μm. A recent report suggests that dislocation density is the major source of strengthening in severe plastically deformed Nb and Ta instead of HP effect [22]. Thus, the role of dislocation density cannot be neglected in understanding the strengthening in SMAT produced microstructure. X-ray line profile analysis becomes an indispensable tool in estimating the average dislocation density and crystallite size in severely deformed metals required to completely understand the strengthening mechanism.

The aim of this investigation is to perform a comprehensive study of the mechanism of nanocrystallization and strengthening in SMAT processed 316L SS. The microstructural characterization was performed at different length scales by TEM, EBSD and X-ray line profile analysis using convoluted multiple whole profile fitting (CMWP).The microstructural parameters like dislocation density, crystallite size, twin boundary frequency, dislocation arrangement and dislocation character were quantified using CMWP. This study reveals the importance of microstructural features like microbands and SBs in the process of nanocrystallization through SMAT. The role of dislocation density and crystallite size in strengthening is also discussed.

2. Materials and methods

2.1. Materials and processing

The starting material for this study was 316L SS obtained in the form of a plate with thickness of 3 mm. The nominal composition of the alloy in wt. % is: Cr 17.20, Ni 11.13, Mn 1.91, Mo 2.5 Si 0.91, P 0.03, S 0.02, C 0.02, and Fe-balance. Specimens were ground up to P 1000 grit prior subjecting to SMAT process. SMAT was performed at a frequency of 50 Hz with 5.5 mm hardened steel balls of hardness 60 HRC for a duration of 15 min. The SMAT equipment had a cylindrical chamber of 80 mm diameter. A total of 100 balls were used in the treatment. The distance between the base of the chamber and the specimen surface was kept at 20 mm.

2.2. X-ray diffraction

The phase constitution and quantification of microstructural characteristics such as dislocation density, dislocation character, dislocation arrangement crystallite size and twinning were evaluated using X-ray diffraction (XRD) patterns. XRD measurements were performed using Cu-Kα radiation (Rigaku Smart Labs) installed with a rotating anode. The measurements were performed at 45 kV and 30 mA current, with a step size 0.02° and a scan speed of 1.5°/min. XRD patterns were recorded at different depths from the surface of the SMAT specimen. The material was removed by etching in an acidic solution to record XRD patterns. A reduction of 10 μm was achieved in every etch cycle followed by XRD