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# Correlation between the surface coverage of severe shot peening and surface microstructural evolutions in AISI 321: A TEM, FE-SEM and GI-XRD study

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ABSTRACT

Severe shot peening (SSP) as a surface severe plastic deformation ($S^2PD$) process over a wide range of coverages was applied to generate gradient microstructures with the grain size increasing from nanometer- and micron-scale to initial grain size on the surface layers of 321 stainless steel. The microstructural evolutions including the grain size distribution and phase transformation were systematically investigated in-depth for different surface coverages. GI-XRD, FE-SEM and TEM techniques were used to reveal the microstructure modification mechanisms as a function of the surface coverage. Experimental results show that dislocation slip plays a key role in the grain refinement of this alloy so that with increasing the surface coverage, different structures including dislocation walls, dislocation tangles, and lamella-shaped cells sequentially appear in the initial coarse grains. The results confirmed that these dense dislocation structures during ultrahigh plastic deformation produce ultrafine- (115–192 nm) and nano-grains (68–82 nm) to minimize the total energy of the system. In line with the grain refinement, the γ (austenite) → α′ (straininduced martensite) phase transformation is more affected as the plastic strain increases. So that the volume fraction of α′ phase increases to 58.4% for ultrahigh strains. Gradient variation of microhardness with the depth was also obtained for various samples.

## 1. Introduction

Corrosion, wear, fretting fatigue and corrosion fatigue are among the major causes of failures in machinery, structures, and equipment. Since these failures originate from the surface, microstructure and properties of the surface layers play a key role in retarding or preventing the initiation of mentioned failures [1–3]. Many researchers claimed that a decrease of the grain size can improve and modify nearly all aspects of the physical and mechanical behaviors of polycrystalline metallic materials as well as their electrochemical and even biomedical responses to the surrounding media [4,5]. In this way, synthesis of ultrafine- and especially nano-grained structures in the surface layers of bulk metallic materials is known as an effective surface modification method for improving the surface properties of different metallic materials such as steel [6,7], aluminum [8], copper [9], nickel [10] and other metals [11,12]. Among different methods, surface severe plastic deformation ($S^2PD$) can be successfully applied to synthesize such microstructures up to about 200 μm below the topmost surface [13].

In recent years, different $S^2PD$ processes including severe shot peening (SSP) [14–16], surface mechanical attrition treatment (SMAT) [17], cold rolling [18], ultrasonic impact peening [19], severe wire brushing [2], high-speed machining [20] and laser shock peening (LSP) [21] have been extensively used to refine the surface grain size by imparting large plastic strains with high strain rates (sometimes around $10^6\ s^{-1}$ [22,23]). Based on the TEM observations, for the above-mentioned processes, a common feature is the creation of high dislocation density areas during the first stages of the process. After imparting further plastic deformation or higher strain rates, more dislocations are created and the structure evolution is followed by recombination and rearrangement of dislocations, and afterwards formation of subgrains with small and/or large misorientations in the initial grains [24].

Since the stacking fault energy (SFE) plays an important role in the deformation process of most metals and alloys, it is more convenient to attribute the grain refinement mechanism of metallic materials after $S^2PD$ processes to this parameter [25]. In general, based on SFE values two different grain structure evolutions can be considered. The first one deals with the materials with higher SFE such as iron (about 200 mJ/m$^2$ [26]) and the second one is related to the materials which have lower

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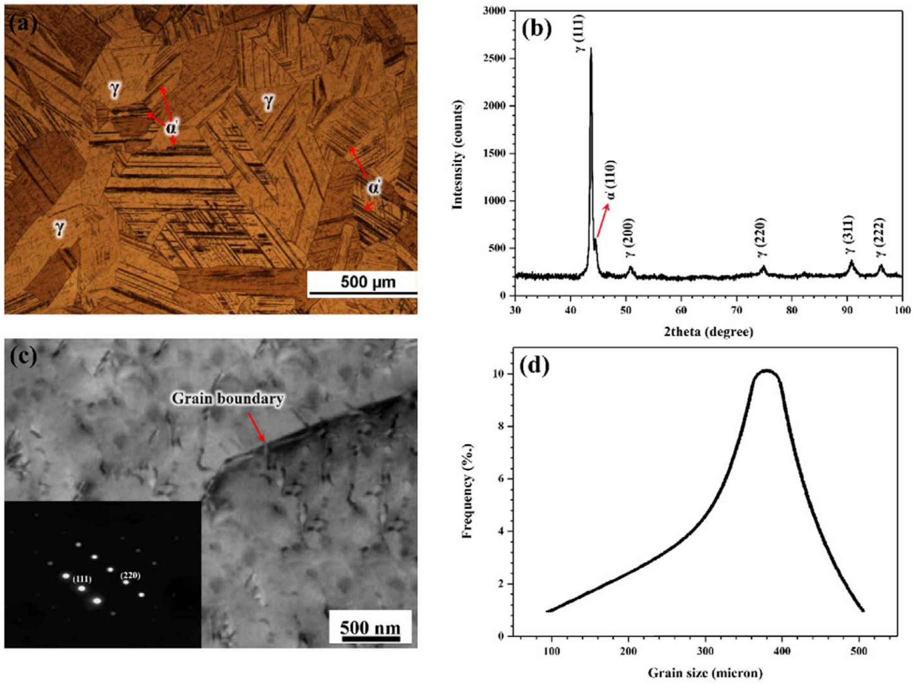


**Fig. 1.** (a) The original microstructure of 321SS alloy before SSP and (b) the corresponding XRD pattern, (c) the bright field TEM image of primary γ phase and corresponding SAED pattern shown as the inset, (d) the grain size distribution of γ phase after solution annealing.

SFE such as austenitic stainless steels (about 78 mJ/$m^2$ [27]). For the first case, the grain refinement is involved in the formation of dense dislocation walls (DDWs) and dislocation tangles (DTs) in the original grains which are subsequently transformed into sub-boundaries and high angle grain boundaries that subdivide the original grains into ultrafine- or nano-grains [28]. On the other hand, in the case of the second grain structure evolution process, original grains are sub-divided by lamellar grains, twins, and micro-twins with nanometer-sized thickness [29]. In both cases, dislocation density and residual stresses are gradually decreased from the topmost surface to subsurface layers, and after reaching a certain depth (dependent on the kind of $S^2$PD process and its intensity, working material, etc.), the grains are completely coarse (just like the surface before the $S^2$PD process).

Among different $S^2$PD processes, SSP which is extensively used in industry has increasingly attracted research interest in the fabrication of nanocrystalline and ultrafine-grains on the surface of bulk materials. Bagherifard et al. [30] reported that nanocrystalline surface layer in cast iron via SSP improved the fatigue strength and crack initiation resistance. Wang et al. [31] produced a nanocrystallized surface layer with an average grain size of 18 nm through SSP on the 1Cr18Ni9Ti stainless steel and pointed out that the chloride-induced corrosion resistance is increased after surface nanocrystallization (SNC). Zhong et al. [32] showed that SNC on the processed surface layer of iron via SSP increased the diffusion coefficient of Al atoms in Fe structure by 4 orders of magnitude.

The aforementioned studies have focused on the grain refinement and nanocrystallization in the top surface and not illuminated the microstructure/grain evolution in the sub-surface layers; however, in-depth microstructural evolutions (especially nanocrystallization and ultrafine-graining) during SSP can be more important than the topmost surface. In fact, this matter should be considered that the surface roughness increment is the perforce change after SSP. Since larger surface roughness can lead to higher stress concentration and worsening in mechanical properties, sometimes the top surface must be ground or polished to achieve a smooth surface. Thus, a thick nanocrystalline/ultrafine layer on bulk metallic materials with a small surface roughness will be the ideal state after SSP.

Hence, knowledge of the in-depth grain refinement of metallic materials during SSP processes helps in better understanding of their microstructure and performance for subsequent applications. In this paper, SSP process with different surface coverages as a $S^2$PD method was applied to fabricate ultrafine-grained and nanocrystalline surface layers on the surface of 321 stainless steel (321SS). Our purpose is to present a systematical study on the effect of surface coverage of shot peening on the in-depth microstructural evolutions in the severely shot peened samples via X-ray diffraction (XRD), field-emission scanning electron microscopy (FE-SEM) and transmission electron microscopy (TEM). The microstructure characteristics including grain size distribution and phase analysis are thoroughly studied in depth. In addition, microhardness variations of the shot peened samples are analyzed.

## 2. Experimental procedure

As-received 321SS alloy bar containing (wt%) 0.021C, 0.557 Si, 1.485 Mn, 18.104 Cr, 0.113 Mo, 9.697 Ni, 0.016 P, 0.011 S, 0.461 Ti and balance Fe was purchased from STOOSS Company. The bar with 80 mm in diameter had been solution annealed at 1100 °C for 2 h. Cylindrical specimens of 6.0 mm thickness were sectioned from the bar and subjected to SSP process using an air blast apparatus (KPS SHOT Co.). For the SSP, standard high carbon steel shots (S230) with a hardness of 45–50 HRC were used. This process was carried out by a peening nozzle with a diameter of 30 mm, mass flow rate of about 8 kg/min, and air pressure of 0.31 MPa. In order to study the in-depth microstructural evolutions as a function of the coverage (shot peening time), SSP processes were performed with different coverages: 200 (conventional shot peening, CSP), 400, 600, 800, and 1000%. According to SAE J442 [33] and SAE J443 [34], these SSP treatments meet Almen intensities between 0.53 mm C and 0.55 mm C. It should be noted that the coverage during shot peening can be related to the peening time. For example, the time needed to reach a coverage of 400% is four times the time needed to reach a 100% coverage (for a given set of peening parameters) [13]. To generate reproducible plastic strains, the shot angle and stand-off distance were set to 90° and 400 mm, respectively. The cross-sections of shot peened samples for FE-SEM observations were mechanically ground, polished and afterward etched in a solution containing 2.5 ml $H_2O$, 2.5 ml $HNO_3$ and 5 ml HCl. FE-SEM images were provided by a TESCAN MIRA3 field emission scanning microscope at 10 kV. The grazing incidence XRD studies were

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