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The role of shear strain on texture and microstructural gradients in low carbon steel processed by Surface Mechanical Attrition Treatment

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ABSTRACT

In this study, the shear strain at various depths of a low carbon steel processed by Surface Mechanical Attrition Treatment (SMAT) was measured using deformed carbide bands as internal strain markers. The shear strain gradient is found to strongly correlate with the gradients of texture, microstructure and hardness. The microhardness increases approximately linearly with shear strain, but deviates at the top surface. In the top surface, the average ferrite grain size is reduced to 60 nm with a strong {1 1 0}//SMAT surface texture.

Gradient structures are known to produce a wide variety of interesting properties including improved wear resistance and fatigue life, and extraordinary mechanical properties [1–6]. In recent years, Surface Mechanical Attrition Treatment (SMAT) has gained attention for its ability to generate gradient structured materials through grain refinement of the surface layer to the nanometer scale [1,7–10]. This technique is ideal for systematic investigations of gradient structures due to the gradients in strain, strain rate, hardness, grain size, and hardening mechanisms throughout the deformed layer. In order to refine grain sizes to the nanometer scale, large strains and strain rates need to be applied [11–13]. It is well known that the shear component of the applied strain is directly correlated with dislocation slip and microstructure evolution. However, quantitatively mapping a single component of the strain tensor is challenging [14–16]. Markers and photographic evidence have been reported to extract the effect of shear strain on microstructure evolution, but they are not suitable for measuring shear strain in SMAT, due to the complexity of the process [17–19].

In this work, cementite bands are used as internal markers to quantify the shear strain at various depths of the surface, which is the first time shear strain has been quantitatively mapped in SMAT-processed samples. Additionally, the texture evolution is systematically characterized, which has rarely been studied in SMAT-processed structures but profoundly affects mechanical behavior [20–23]. The observations from this work elucidate the effect of shear strain on the development of texture gradient, microstructure gradient, and microhardness gradient in the SMAT-processed samples.

Normalized steel plates with a composition of 0.14% C, 0.33% Si, 1.44% Mn, 0.08% Cr, 0.03% Ni, and balance Fe was used for this study. Samples were cut along the rolling direction so that the SMAT treatment would take place normal to the rolling direction. The pearlite was agglomerated into bands normal to the SMAT surface as shown in Fig. 1. The SMAT process was carried out using a SPEX 8000M Mixer/Mill by replacing the lid of the vial with 1/4″ thick plates of the sample to be treated. Samples were polished to 1200 grit, sealed in ambient atmosphere, and processed with three ½″ 440C steel balls for 120 min. Profilometry revealed that the surface was roughened to an Ra of 8.8 μm. After treatment, cross-sectional samples were Ni-plated to protect the surface microstructure from edge rounding when polishing, and were imaged using a JEOL 6010LA Scanning Electron Microscope (SEM) at 20 kV.

Experimentally measuring a single component of the strain tensor is not a trivial matter. Attempts to discern the shear component

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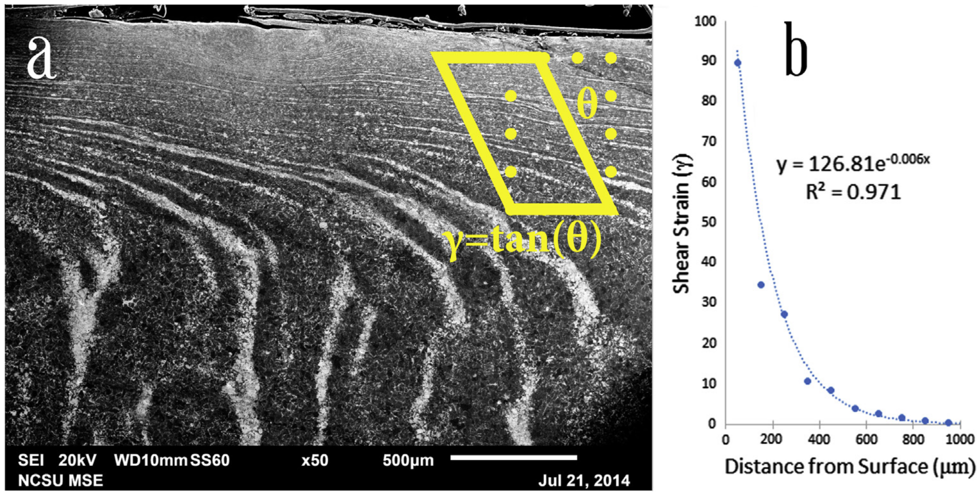

Fig. 1. (a) An SEM image of the as-received SMAT surface. The inset shows how the slope of the cementite bands was measured in order to calculate shear strain γ. (b) The resulting data calculated at various depths showing a clear exponential increase in shear strain approaching the surface of the SMAT gradient.

of the tensor in severe plastic deformation processes have been undertaken primarily by simulation or calculation [11,14,16,18]. Experimentally, an imbedded pin method has been used to measure shear in accumulated roll bonding, where the interface of the pin can be used to map the shear strain, but this is not suitable for SMAT structures [19]. In normalized low carbon steels, clear bands of pearlite along the rolling direction (α-iron + Fe_3C) and ferrite (α-iron) naturally act as markers in the microstructure. Since the plates were cut normal to the rolling direction, these bands were perpendicular to the SMAT surface, as shown in Fig. 1. Because the shot impacts from SMAT repeatedly induce plastic flow in the surface, the top deformed layer is theorized to undergo high shear strains [1,17]. Therefore, simply mapping the slope of the deformed pearlite bands could yield the accumulated shear strain induced at various depths of the SMAT surface. First, a grid with 100 μm blocks was overlayed onto the micrograph seen in Fig. 1A. Then, the average slope was measured in each of these regions to calculate the average shear strain at various depths. In this way, the shear strain plotted at 50 μm, 150 μm, 250 μm, etc. represents the average shear strain at each 100 μm interval. The result indicates that the shear strain increases exponentially near the surface, which is visually apparent in the cross-sectional SEM micrograph (Fig. 1A). A simple exponential fit ($R^2 = 0.97$) was applied to the data in order to estimate the shear strain at discrete depths from the surface. Extrapolation of this data was used to estimate the shear strain at depths less than 100 μm but it is not clear how big the error is from such an extrapolation (Fig. 2). Surprisingly, the average measured shear strain at a depth of 50 μm is 90, and the extrapolated shear strain in the top 10 μm is 119, which is in the realm of shear strains measured in accumulated roll bonding and chip processing, as well as high pressure torsion [14,18,19]. Note that in the very top surface, e.g. at layer thickness close to the roughness, the current strain measurement may significantly underestimate the shear strain.

Once the shear strain was calculated, grain size and microhardness measurements could be plotted to determine their relationship. Five hardness measurements were averaged at each depth with a Mitutoyo Microhardnss Testing Machine Model HM-11 with a Vickers diamond indenter at a load of 0.05 N. Grain size measurements were performed using the line intercept method from micrographs collected from the dual Beam FEI Quanta 3D FEG, the JEOL 2010 F Transmission Electron Microscope. Fig. 2 shows these relationships and a corresponding FIB micrograph of the gradient structure at the surface. Both hardness and grain size show a strong dependency on the shear strain, and the Hall–Petch plot shows slight deviation from the ideal linear trend.

At the top surface, the grain size is dramatically reduced to 60 nm, as seen in Fig. 3. TEM samples confirmed that the grain size at the top surface was skewed, with some regions containing grains less than 10 nm, while other regions had grains 100 nm in diameter. Fig. 3 shows the distribution of grain size at the top 10 μm of the SMATed surface. In carbon steels, nanocrystallization has been reported in regions subject to very high shear strains, and were first discovered in railroad tracks [14]. These regions were called "white etching layers", which consist of fragmented Fe_3C and even complete Fe_3C dissolution that leads to supersaturation of carbon in nanocrystalline α-iron [6,15,24]. These reports are consistent with this observation here.

Because SMAT is a complex deformation scheme consisting of compressive and shear strains at various strain rates, mapping the texture allows for a simple investigation on the underlying deformation schemes at various depths from the surface [1,23]. Samples were prepared for EBSD imaging by conventional polishing followed by ion milling in a Fischione Ion Mill (Model-1060) at 5 kV and 5° tilt for 45 min. An Oxford EBSD detector installed in the dual Beam FEI Quanta 3D FEG was used for collecting images. Fig. 4 shows an overview of the microstructural and textural development along the depth. The as-received material consists primarily of high-angle grain boundaries (>15°) and displays no strong texture. After SMAT, at 200 μm below the surface, no strong texture can be determined, but the grain size has been reduced and clear subgrain boundaries (>2°) can be seen within large grains. At 100 μm below the surface, there is a clear transition to a complex texture with {110} and some {111} planes//SMAT surface, and low-angle grain boundaries have evolved from the subgrain boundaries. At 50 μm below the surface, although some residual {111}//SMAT surface texture can still be seen, the texture mostly transitioned to {110}//SMAT surface, which is a well-known texture for highly sheared α-iron [21,23]. At the top surface, the {110}//SMAT surface texture is further strengthened, and most grain boundaries have been converted to high angle. As can be seen in Fig. 4, the development of the texture of {110}//SMAT surface is preceded by diminishing {111} components from the depth of 100 μm to the surface.

Texture develops when preferred crystallographic orientations align with applied stress. Slip systems tend to align with the shear direction to maximize the resolve shear stress [1,22]. In BCC α-iron,

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