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Development of nanocrystalline structure in Cu during friction stir processing (FSP)

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ABSTRACT

The characteristics of microstructures at various locations behind the pin tool extraction site were studied in copper after FSP that had been conducted with continuous quenching to enhance cooling rates. The substructures initially formed around the pin tool consist of very small crystallites having sizes of a few tens of nanometers. It is proposed that the processing conditions result in formation of microband structures around pin tool in the presence of severe strain heterogeneity. The microbands appear as nano-scale elongated crystallites surrounded by high-angle boundaries. The elongated crystallites transform to nearly random oriented and equiaxed grain structures by shape adjustment during the initial stages of cooling from the peak temperature. Nanocrystalline structures ~174 nm in size were produced in OFHC copper by FSP.

1. Introduction

Many methods for grain refinement and property enhancement in metals and alloys include plastic deformation of the material [1]. Two conditions should be met in order to produce nanocrystalline structures by plastic deformation: (a) a large concentration of nuclei or sub-structures of nano-scale size are formed in the deformed material, and (b) the nuclei or sub-structures can transform to new grains surrounded by high-angle boundaries without significant grain growth. It is usually thought unlikely that nano-scale grain sizes can be attained in metals and alloys by high-temperature plastic deformation because of the difficulty in attaining a sufficient concentration of refined sub-structure units as well as a strong growth tendency of such units as they transform from sub-structures to new grains at elevated temperature.

Friction stir processing (FSP) is a recently developed thermo-mechanical processing technique based on the principles of friction stir welding (FSW) [2,3]. The material undergoes intense plastic deformation at temperatures near the peak temperature of the FSP/FSW thermo-mechanical cycle. The local peak temperatures attained in various aluminum and copper alloys have been found to be between $0.6T_m$ and $0.95T_m$, values which correspond to the hot working temperature range for these metals and alloys [4].

Conventional thermo-mechanical processing methods, e.g., hot rolling, involve approximately uniform and isothermal straining of work piece materials. In contrast, the FSP/FSW thermo-mechanical cycle involves rapid transients and steep gradients in strain, strain rate and temperature. During FSP the material in contact with the pin tool experiences the largest strain, the highest strain rate and attains the highest peak temperature. The resulting microstructures then evolve after passage of the pin tool in a region of decreasing strain, strain rate and temperature.

By combining enhanced cooling methods with FSP, nanocrystalline structures have been created in Al alloys [5–7]. However, the mechanisms governing the formation of such small grains were not established. Restoration mechanisms such as dynamic recovery or recrystallization that are understood to act during conventional thermo-mechanical processing are inadequate to explain refinement to nanoscale level during FSP of these alloys.

To establish the mechanisms of formation of nanocrystalline structures during FSP it is necessary to examine the sub-structures formed in earliest stages of deformation nearby the pin tool. Orientation imaging microscopy (OIM) can provide quantitative measurement of grain structures at such locations and reveal microstructure evolution by evaluating grain characteristics in locations behind pin tool. Such information is difficult to obtain in highly refined structures in Al alloys but good quality elec-

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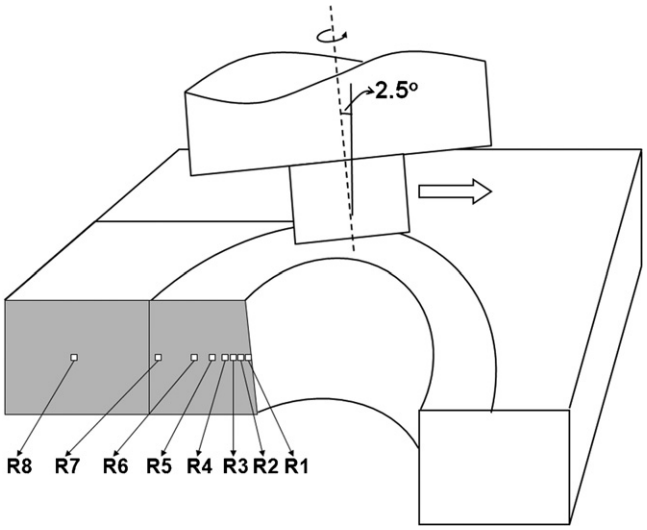

Fig. 1. Schematic the FSP and the positions scanned by OIM in the processed material.

tron backscattered diffraction (EBSD) patterns allow OIM for grains smaller than 100 nm in copper.

The production of nanocrystalline structures during conventional high temperature deformation is generally not feasible even in alloys with multiple solute impurities and various particles to retard grain growth. It would likely be impossible in pure metals. In the present work, FSP was performed on commercial OFHC (C10100) copper with a purity of 99.99% and practical measures were employed to enhance cooling rates to preserve the deformation-induced features of microstructure. Then, microstructures at the pin tool surface as well as in various locations behind tool were examined to assess the formation and evolution mechanisms of nanocrystalline structures during FSP.

2. Experimental

Polycrystalline copper sheet 2 mm in thickness was prepared by cold rolling and annealing of a commercial OFHC (C10100) copper plate having a purity of 99.99%. The initial Cu plate thickness of 6 mm was reduced to 2 mm in three rolling passes with 30 min anneals at 500 °C after each rolling pass, providing a final annealed microstructure with a grain size of ~70 μm. A smooth FSP tool with shoulder diameter, pin diameter and pin length of 7.5 mm, 2.5 mm and 2 mm, respectively, was fabricated in H13 steel and hardened to HRC 52. The tool was tilted ~2.5° opposite the traveling direction during a single processing pass on the Cu sheet at 800 rpm and a travel speed of 120 mm min^{-1}. Dry ice was continuously placed on the surface of the processed material behind the tool to enhance cooling rates during the processing run. The pass was terminated prior to reaching the edge of the sheet by quickly lifting the rotating tool and the material surrounding the tool extraction site was then immediately quenched with additional dry ice in order to preserve the microstructures at locations around the tool. It should be mentioned that because of tilting of the tool, the material behind the tool quickly detached itself from the tool pin and remains intact during the rapid tool extraction, whereas the material ahead the tool might be pulled out with the lifted tool.

The as-processed sheet was sectioned through the pin extraction site along the axis of the tool traverse in order to reveal the plane defined by the traversing direction and the sheet normal, as indicated in Fig. 1. Samples for OIM analysis were ground on progressively finer SiC papers to a 4000 grit finish and then polished with 0.05 μm colloidal silica in a vibratory polisher for 6 h. After two weeks of the FSP, grain structures of the FS processed sample were examined using an EDAX-TSL OIM system installed on a Philips XL-30S FEG scanning electron microscope. The scanning step size corresponds to a pixel size of 10 nm × 10 nm and was used for all regions behind pin tool. The error in the determination of the crystallographic orientation of each grain was less than 2°.

A larger area OIM scan was performed to assess the final microstructure for statistical purposes. In this scan, the scanning step was 20 nm and the resulting data were subjected to clean-up procedures as follows: (1) grain dilatation with a grain tolerance angle of 5°, and (2) each grain contains at least two scan points.

3. Results and discussion

Microstructures at the mid-depth of the stir zone and various locations behind the tool pin were examined by OIM. As illustrated in Fig. 1, these regions are denoted R1–R8, and are at progressively greater distances from the tool pin–stir zone interface. Locations R1-R7 were under the tool shoulder, and are ~0, 0.01, 0.02, 0.06, 0.2, 1 and 2 mm away from the tool pin, respectively. Location R8 is located beyond the tool shoulder and exhibits essentially the final state of the microstructure.

3.1. Microstructure formed around pin tool

The OIM inverse pole figure (IPF) map in Fig. 2a is from location R1 (Fig. 1) and shows the highly refined crystallites present nearby the tool pin–stir zone interface at the pin extraction site. These substructures were formed in the condition of peak temperature, largest strain and highest strain rate during FSP and comprise very small elongated crystallites with widths of 20–70 nm and lengths of 50–200 nm. The boundary statistics of these crystallites are presented in the misorientation angle distribution in Fig. 2b. The crystallite boundaries are mainly of high-angle in nature. Thus, the substructures formed around pin tool are nano-scale crystallites surrounded mostly by high-angle boundaries.

Metals and alloys of high stacking fault energy (SFE) undergo restoration by dynamic recovery (DRV) when subjected to deformation at elevated temperature while those having low or medium SFE, such as copper [8], exhibit restoration by DRV and/or dynamic recrystallization (DRX). Derby [9] has further classified DRX mechanisms into rotational and migrational types. In migration recrystallization, new strain-free grains nucleate and grow within the deforming material when the strain reaches a critical level. In rotation recrystallization, a dynamically recovered substructure undergoes a gradual increase in misorientation, leading to the transformation of cell boundaries into sub-boundaries, and then into high-angle boundaries.

Although it is unlikely that nanocrystalline structures will form in metals and alloys through DRX in conventional thermomechanical processing, very fine grained structures 0.1–0.3 μm in size have been reported in Cu subjected to high-strain and high-strain-rate deformation, e.g., after shock loading [10,11]. The peak temperature attained during such loading was estimated to be $0.4T_m$–$0.5T_m$, at which the recrystallization (static or dynamic) occurred, when the material was initially at room temperature. Dynamic recrystallization was proposed to be responsible for the refined grain structures. The evolution of the deformation-induced microstructure during such high-strain, high-strain-rate deformation was described as: (1) elongated dislocation cell formation by dynamic recovery; (2) formation of elongated subgrains; and (3) break-up of elongated subgrains to form a microcrystalline structure [10,11]. The present results, as shown in Fig. 2, indicate that very refined substructures form around pin tool during the FSP. The possibility that these substructures are dislocation cells formed by