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In the search of nanocrystallinity in tool-steel chips

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Abstract

Proper waste handling and sustainable recycling approaches are emerging topics for environmental safety and ecomanufacturing technologies. Steel chips resulting from new machining procedures are good candidates as new raw material for innovative recycling approaches due to their unique characteristics. The objective of this study was to characterize as-quenched H13 (AISI-SAE) tool steel chips produced by a machining procedure. Microhardness tests, SEM/EBSD and TEM techniques were used for hardness evaluation and microstructural characterization. Chips display a highly strained submicron-nanocrystalline grain structure with hardness up to 6.2 GPa. Such characteristics suggest the chips are suitable for powder manufacturing processes such as ball milling instead of atomization process.

Keywords: Tool steel (H13); chips; waste; machining; ecomanufacturing; recycling.

1. Introduction

Steel plays yet an important role in world's economy. Subtractive manufacturing processes, such as steel machining and finishing, produce enormous quantities of metallic chips every year. Proper waste handling and new sustainable recycling approaches are emerging topics for environmental safety and ecomanufacturing technologies. Typical mild steel machining chips are regarded and disposed as metal scrap, dispatched for remelting in steelmaking industries. Advances in machining procedures allow the processing of hard grade steels such as as-quenched H13 (AISI-SAE) [1-3]. Metallic chips resulting from hard grade steels attracted considerable attention since they are expected to exhibit unique microstructural characteristics such as nanocrystallinity. Could it be, what is typically considered valueless waste, regarded as an excellent raw material for innovative recycling approaches? Taking advantage of unique steel chips characteristics, we can add them great value, as powders that are a high-cost material [4]. The objective of this study was to characterize as-quenched H13 tool steel chips produced by a machining procedure.

2. Experimental

The metallic chips used in this work were obtained by a machining procedure of an H13 (AISI-SAE) tool steel in the quenched condition (Vickers hardness ≈ 5.4 GPa). Cutting parameters used are shown in Table 1. Chips were collected and dispatched for size and morphology evaluation by stereomicroscopy (SM) and scanning electron microscopy (SEM). X-ray diffraction was performed on the as-machined chips (Co-K_α = 1.79026). Microstructural characterization was performed after specimen's preparation by means of mounting, grinding and fine-polishing the chips, for optical microscopy (OM) and electron backscatter diffraction (EBSD), and by focused ion beam (FIB) for transmission electron microscopy (TEM). Chip hardness was evaluated by Vickers microhardness tests.

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3. Results and Discussion

Chips were observed as received by stereomicroscopy and by SEM (Fig. 1). The high hardness of the metallic workpiece promotes the breaking down of the chip into small comma-like shaped pieces, with high aspect ratio, < 10 mm long, < 1 mm wide and < 0.1 mm thick.

Table 1. Machining parameters used on H13 workpiece.

Machining parameters	
Cutting speed (m/min.)	90
Cutting depth (mm)	0.1
Feeding speed (m/min.)	3
Cutting tool rotation (rpm)	2000
Medium	Air

Sharp-cut edges separate two different surface morphologies. The side of the chip contacting with the cutting tool is smooth and shiny, whereas the opposite surface is rough and irregular. The heat produced by friction between the chip and the cutting tool and by straining generates a thin oxide film covering both chip surfaces in different colours, from brown-purple to pale blue, suggesting the chip achieved temperatures up to 260-320ºC [5]. The removal of such oxide film is of paramount importance if the chips are to be used for powder manufacturing. This step is accomplished during the manufacturing of powders.

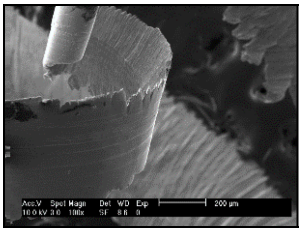


Fig. 1. Chips as received observed by SEM.

The microstructure observed on chip longitudinal-section reveals a fine dispersion of carbides (likely chromium and vanadium) embedded in an elongated-grain matrix [6] (Fig. 2). Vickers microhardness tests, performed on the chips was 6.22 ± 0.08 GPa, indicating a strained martensitic grain structure [7]. XRD results (Fig. 3) show, by Scherrer's equation, that the crystalline dimension is approximately 12 nm. Bright field TEM images performed on a thin focused ion beam cut section reveal a strained grain structure (Fig. 4). Sharp grain boundaries are not easy to identify but electron diffraction patterns, consisting of broken rings (Fig. 4 a)), suggest heterogeneous crystallographic orientations for the same lattice plane. This means that either high-angle grain boundaries (HAGB), low-angle grain boundaries (LAGB), or a mixture of both can be present. Such boundaries are due to dislocation stacking formed by the heavy straining [8,9]. A sharp spot diffraction pattern indicates a single crystal carbide particle (V_xC_{x-1}) (Fig. 4 b)).

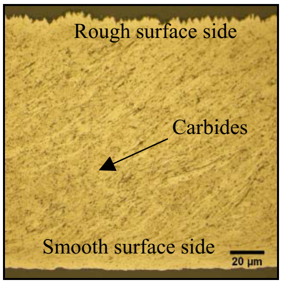


Fig. 2. Microstructure of a chip longitudinal-section. Elongated grain structure with precipitate dispersion.

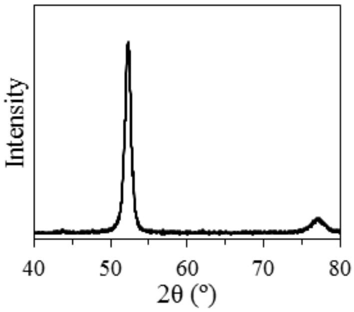


Fig. 3. XRD diffractogram of chips.

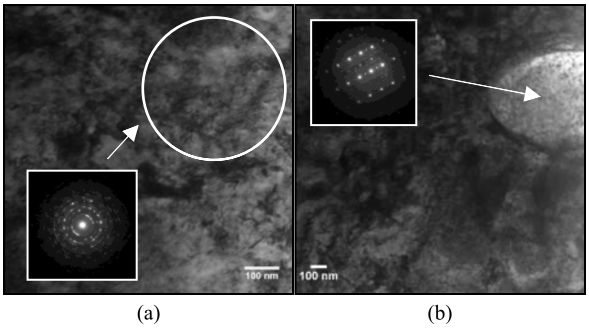
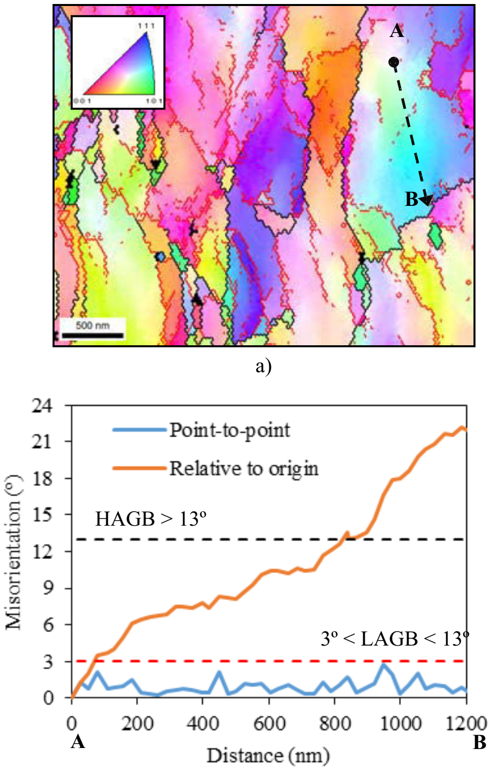


Fig. 4. Bright field TEM images and diffraction patterns of chip microstructure.

EBSD analysis also suggests a highly strained grain structure (Fig. 5 a)). Colours indicate crystallographic orientation relative to the normal of the analysed surface, according to the legend. Black lines represent HAGB > 13º, whereas red lines designate LAGB, between 3 and 13º. Microstructure displays several LAGB within elongated submicrometric grains [10] according to XRD and TEM results. Moreover, cumulative crystallographic rotation (< 3º increment), given by hue variation within a grain, indicates a stacking of a high number of crystal defects such as dislocations. For instance, one can consider the distance between two points within the same grain, highlighted by the black arrow (Fig. 5 a)). The chart in Fig. 5 b) plots the misorientation measured between point A (origin) and point B. Although the point-to-point (30 nm step) misorientation is below 3º, it accounts for a total rotation of nearly 23º along a distance of 1.2 µm, a cumulative lattice misorientation introduced by dislocation stacking.



a)

b)

Fig. 5. EBSD inverse pole figure map with HAGB in black and LAGB in red (a), and bright crystal misorientation profile between points A and B (b).

4. Conclusions

Steel chips from as-quenched H13 tool steel machining display a high aspect-ratio shape. A highly strained submicron/nanocrystalline martensitic grain structure, with hardness around 6.2 GPa, makes these chips suitable for powder production by ball milling in a reducing atmosphere. Nevertheless, it is necessary to have a better understanding of the grain refinement mechanism.

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