Small-angle neutron scattering investigations of nanocrystalline alloy chips obtained by machining

Elwyn Rebello, Frazer Vaz, Avino Colaco, Wellon Rodrigues, Geethalakshmi K, Debasis Sen, S. Mazumder and A.O. Surendranathan

Cogent Engineering (2014), 1: 951149
Small-angle neutron scattering investigations of nanocrystalline alloy chips obtained by machining

Elwyn Rebello¹, Frazer Vaz¹, Avino Colaco¹, Wellon Rodrigues¹, Geethalakshmi K²*, Debasis Sen², S. Mazumder² and A.O. Surendranathan³

Abstract: Ultrafine-grained (UFG) materials exhibit significantly enhanced mechanical properties. This has brought renewed attention on the use of large strain or severe plastic deformation as a means for achieving microstructural refinement in metals and alloys. Large plastic strains imposed in a machine chip result in significant microstructural refinement, including the creation of UFG and nanocrystalline materials. It looks to be an economical route for realizing nanocrystalline materials. In the present study, small-angle neutron scattering (SANS) was employed to investigate the modifications in the microstructure of the chips produced via machining. Double crystal-based medium resolution SANS instrument has been used for this purpose. Significant scattering intensity at small enough angles reveals the presence of mesoscopic density fluctuations produced because of the machining. Atomic force microscopy images also corroborate the existence of such small length scale density fluctuations.

1. Introduction

It has been proved by time and again by researchers that nanocrystalline materials have enhanced mechanical properties in terms of hardness and strength over the bulk materials. However, the production cost of nanocrystalline materials is excessively high due to the equipment used and the probable low output. Recently, researchers have proved that nanocrystalline materials can be produced through machining (Ni & Alpas, 2003; Shankar, Chandrasekar, Compton, et al., 2005; Shankar, Chandrasekar, King, et al., 2005; Swaminathan et al., 2005). They have demonstrated that under certain combinations of machining parameters, large shear strains can be imposed in the machining
process, resulting in nanoscale microstructures. The availability of large volumes of metal chips, as bi-products either from existing manufacturing operations or from specific chip-making processes, offers opportunities to create nanocrystalline materials with mechanical properties that are influenced by the ultrafine-grained microstructure of the chips. Furthermore, the production costs for nanocrystalline materials created using the machining-based deformation processes are much less than those of conventional methods (Iglesias et al., 2008). However, this extremely fine microstructure is difficult to image without the use of Transmission electron microscopy (TEM) or Field emission-Scanning electron microscopy (FE-SEM).

Small-angle neutron scattering (SANS) can be used to determine the shape and the organization, averaged in time, of particles or aggregates dispersed in a continuous medium (Borsali & Pecora, 2008). The term particle is applied to a wide range of objects from the fraction of a nanometer to the micrometer level. The use of large samples without any special preparation and of a non-destructive character gives it an edge over other techniques, like TEM, FE-SEM, Field ion microscopy, etc.

In the present work, machined chips were produced from two commercially available and frequently used alloys through orthogonal machining. Investigations were carried out on the chips through SANS along with Scanning electron and Atomic force microscopy (AFM), followed by mechanical characterization through shear strain and microhardness testing.

2. Experimental

2.1. Materials
Two commonly used alloys, viz. commercially available steel and brass with respective near equivalent specification IS 45C8 and IS 319/74, were used for the present investigation, keeping in view of the fact that these alloys are most often shaped through regular machining operations. The nominal chemical compositions of these two alloys are given in Table 1.

2.2. Machining
Machining chips were produced by using with high-speed steel and carbide tools under controlled plain strain conditions with a range of rake angles and machining speeds. Feed rate was kept at .2 mm/rev and rake angle was varied from +8° to −15° with a depth of cut of 40 μm. The machining velocity was kept at 15–20 mm/s to avoid the influence of temperature. The initial grain sizes of the bulk material in both cases were greater than 10 μm. The bulk hardness of steel was ~140 HV and

| Material | Composition (wt %) |
|----------|-------------------|
| Steel    | Element C Si Mn P S Cr Ni Mo Al Cu Fe |
|          | % .463 .277 .81 .016 .02 .177 .016 .02 .03 .01 Balance |
| Brass    | Element Zn Pb Sn Fe Ni As Sb Bi Ag Co Cu |
|          | % 37.82 2.8 .38 .259 .157 .17 .048 .018 .023 .024 Balance |

Figure 1. Schematic of large strain machining.
that of brass was around ~110 HV. Typical chip samples were 50–80 μm in width, 40–60 μm in thickness, and 100–200 μm in length.

During the cutting process, the tool and work piece are forced against each other and a compressive force is set up, which causes the metal to deform in front of the tool point. The deformation takes place in a zone along the shear plane and the metal is forced to slide over the tool surface. In doing this, the shear stress causes the material to separate as a chip, based on the rake angle. With positive rake angle, the area under shear decreases leaving less deformation on the surface of the work piece (Geels, 2007). A schematic view of large strain machining is shown in Figure 1.

From the measured deformed chip thickness (ac) and undeformed chip thickness (ao) and the shear plane angle (ϕ), the shear strain values (γ) imposed in the chip were estimated using Equations 1 and 2 (Merchant, 1945; Shaw, 1984).

\[ \tan \varphi = \frac{\frac{a_o}{a_c} \cos \alpha}{1 - \frac{a_o}{a_c} \sin \alpha} \]  
\[ \gamma = \frac{\cos \alpha}{\sin \varphi \cdot \cos(\varphi - \alpha)} \]

2.3. Characterization

The chips were characterized by SANS for estimation of crystallite/particulate size followed by the determination of Vickers microhardnesss and microstructural characteristics. Microhardness measurements were done on a Matsuzawa Micro Vickers Hardness Tester Model MMT-X7. SANS measurements were performed using a double crystal-based medium resolution SANS instrument (Mazumder, Sen, Sarvanan, & Vijayraghavan, 2001a, 2001b) at the guide tube laboratory of DHURVA reactor, India. The scattered intensities \([I(q)]\) have been recorded as a function of wave vector transfer \(q = \frac{4 \pi \sin(\theta)}{\lambda}, \) where \(2\theta\) is the scattering angle and \(\lambda (=0.312 \text{ nm})\) is the incident neutron wavelength for the present experiment. The specimens were placed on a sample holder with a circular slit of 1.5 cm diameter. The SANS data were corrected for background, transmission, and instrument resolution effects (Lake, 1967; Schmidt, 1988). Microstructural characterizations were done using an optical microscope, JEOL Scanning Electron Microscope, operating at 20 kV and NTEGRAPRIMA/NTMDT Atomic Force Microscope. For metallographic observation, the as-machined chips were collected and mounted in a resin and mechanically polished with wet SiC paper of 2000 grit and 3000 grit and then finely polished with .05 and .02 μm Al₂O₃ powder, followed by chemical etching.

3. Results and discussion

3.1. Morphology and microstructure

The optical micrographs of the bulk specimen are shown in Figure 2, with (a) and (b) representing the respective microstructures of steel and brass in the bulk state. The average grain size of steel specimen is ~15 μm, while that of brass is ~25 μm.

The representative of scanning electron micrographs of the as-machined steel chip at different magnification is shown in Figure 3. Figure 3(a) represents the SEM micrograph of typical cut chip specimen exhibiting a “flow-line” type microstructure, characteristic of large strain deformation. Figure 3(b) and (c) represent the cross section of chip segments that are attached loosely to each other, exhibiting intense shear localization and formation of micro shear bands. There exists a distinct direction of shearing throughout the chip cross section and a thin secondary shear zone along the back of the chip. These shear bands are formed due to high strain rate and large plastic deformation.

AFM could be a faster alternative to TEM techniques for trying to image the microstructure within shear bands. Figure 4 represents the atomic force micrographs of metallographically prepared specimens. Figure 4(a) is a typical AFM 2D image of the machined chip prepared by embedding the chips
in a polymer matrix. The image reveals the distinctive shear bands that are formed during machining as mesoscopic traces, aligned with the shear plane. Figure 4(b) shows the microstructure of brass chip having an average grain size of ~40 ± 10 nm, while Figure 4(c) shows that of the steel chip having an average grain size of ~60 ± 10 nm. The 3D image (Figure 4(d)) shows the average height and roughness of the chip surface.

3.2. Shear strain and hardness
The shear strain values in the chip as estimated from Equations 1 to 2 were found to vary from 1 to 3.9 with a change of rake angle from +5° to −15°. The dislocations that are generated to accommodate these substantial strains assemble themselves into complex and elaborate cellular structures in the chip (Shekhar, Cai, Lee, Wang, & Shankar, 2009) leading to the formation of deformation shear bands. The Vickers hardness values of the chips were determined with a load of 300 g on steel and 200 g on brass samples. The Vickers hardness values of the chips machined at −5° and −15° rake angle were found to be 235±5 and ~256±5HV, respectively, and that of brass were found to be 175±5 and 198±5HV, respectively. So, the machined chips exhibit a remarkable increase in the hardness as compared with the measured bulk hardness values of the two materials. This increase in hardness values is undoubtedly a consequence of the reduced grain size and dislocation substructures in the chips.
3.3. Small-angle neutron scattering
Small-angle scattering signal originates from the density fluctuations in the mesoscopic length scale. In the present case, it is assumed that the scattering intensity at small angle, as observed experimentally, originates from the density fluctuation because of the presence of the sub-micrometric...
structure after machining. It is observed from the scattering profiles that the scattering profile becomes significantly broader due to small-angle scattering after the machining. The scattering intensity \( I(q) \) at small-angle region corresponding to polydisperse pores may be approximated as:

\[ I(q) = C \int_0^\infty D(R) R^6 P(q, R) S(q, R) dR \]  

where \( D(R) \) represents the size distribution of the basic constituents of the fractal, i.e. \( D(R) dR \) represents the probability of finding a particle with radius \( R \) to \( R + dR \). \( P(q, R) \) is the form factor of a particle of radius \( r \). In present case, a spherical form factor is assumed for the data analysis,

\[ P(q, R) = 9 \left( \frac{\sin(qR) - qR \cos(qR)}{(qR)^3} \right)^2 \]  

\( C \) is the scale factor and is independent of \( q \), \( S(q, r) \) is the interparticle structure factor. A fractal like \( S(q, r) \) was considered in the present case to take into account of the particle cluster. The neutron scattering profiles of the machined chips are presented in Figure 5.

The above model was fitted to the experimental profile and the particle size distribution was estimated using nonlinear least-square method.

The estimated particle size distribution for the machined samples is plotted in Figure 6. The average radius of the grains in machined samples is nearly 45 nm. It was found that a consideration
of a fractal-like structure factor, due to the clustered particles, fits the data fairly well. The upper cutoff of the fractal was found to be above 300 nm, although no distinct value could be obtained due to low q limit. From the analysis of scattering data, it was found that for the un-machined sample, the grain sizes are large enough (>1 micron) compared with the grain size in the machined samples, but no conclusive decision about the grain size of the un-machined sample could be drawn as the larger grain size demands an access to further low q values.

The average particle size obtained from USANS experiment is found to be in fair corroboration with the grain size as obtained from AFM. It is to be mentioned that the grain morphology obtained from AFM represents nearly coherent arrangement within the local zones, whereas the particle size obtained from SANS represents the average size of the primary particles of the aggregates in the machined chip. It should be mentioned that the presence of mesoscopic traces of shear bands in the machined chips, formed due to large strain deformation, may also lead to the density fluctuations as far as scattering data are concerned.

4. Conclusion
Fine microstructure, composed of grains even smaller than 100 nm, is found to result from chip formation in commercial steel and brass due to the induced shear strain leading to large strain
formation. The machined chips exhibit a remarkable increase in the hardness as compared with
the measured bulk hardness values in both materials. This increase in hardness values is attributed
to the reduced grain size and dislocation substructures in the chips. The average particle size obtained
from neutron scattering experiment is found to be in corroboration with the particle size obtained
from AFM. Furthermore, the presence of mesoscopic traces of shear bands in the machined chips
formed due to large strain deformation leads to the density fluctuations during neutron scattering.

Funding
The authors received no direct funding for this research.

Author details
Elwyn Rebello1
E-mail: elwynreb66@gmail.com
Frazer Vaz1
E-mail: frazervaz@gmail.com
Avino Colaco1
E-mail: avino.colaco@yahoo.com
Wellon Rodrigues1
E-mail: wellor@yahoo.com
Debasis Sen1
E-mail: geethashyam@gmail.com
Geethalakshmi K1
E-mail: wellor@yahoo.com
A.O. Surendranathan3
E-mail: aos_nathan@yahoo.com
S. Mazumder2
E-mail: smazu@barc.gov.in
A.O. Surendranathan, Cogent Engineering (2014), 1: 951149.

References
Borsali, R., & Pecora, R. (Eds.). (2008). Soft-matter characterization: Small-angle neutron scattering and
applications in soft condensed matter (Vol. 2). New York, NY: Springer Science + Business Media LLC.
Geels, K. (2007). Metallographic and materialographic specimen preparation, light microscopy, image analysis and hardness
testing. West Conshohocken, PA: ASTM International.
http://dx.doi.org/10.1520/MNL46-EB
Iglesias, P., Moscoso, W., Mann, J. B., Saldana, C., Shankar, M. R., Chandrasekar, S., ... Compton, W. D. (2008).
Production analysis of new machining-based deformation processes for nanostructured materials. International
Journal of Material Forming, 1, 459–462. doi:10.1007/s12289-008-0094-0
Lake, J. A. (1967). An iterative method of slit—Correcting small angle X-ray data. Acta Crystallographica, 23, 191–194.
doi:10.1107/S0365110X67000240
Mazumder, S., Sen, D., Sarvanan, T., & Vijayraghavan, P. R. (2001a). A medium resolution double crystal based small-angle
neutron scattering instrument at Trombay. Current Science, 81, 257–262.
Mazumder, S., Sen, D., Sarvanan, T., & Vijayraghavan, P. R. (2001b). Performance and calibration of the newly
installed medium resolution double crystal based small angle neutron scattering instrument at Trombay. Journal of
Neutron Research, 9, 39–57.
doi:10.1080/102381601018200241
Merchant, M. E. (1945). Mechanics of metal cutting process 1: Orthogonal cutting and type 2 chip. Journal of Applied
Physics, 16, 267–275.
http://dx.doi.org/10.1063/1.1707586
Ni, H., & Alpas, A. T. (2003). Sub-micrometer structures generated during dry machining of copper. Materials
Science and Engineering A, 361, 338–349.
doi:10.1016/S0921-5093(03)00530-6
Schmidt, P. W. (1988). Collimation effects in small-angle X-ray and neutron scattering. Journal of Applied Crystallography,
21, 602–612. doi:10.1107/S0021889888006375
Shankar, M. R., Chandrasekar, S., Compton, W. D., & King, A. H. (2005). Characteristics of aluminum 6061-T6 deformed to
large plastic strains by machining. Materials Science and Engineering A, 410–411, 364–368.
doi:10.1016/j.msea.2005.08.137
Shankar, M. R., Chandrasekar, S., King, A. H., & Compton, W. D. (2005). Microstructure and stability of nanocrystalline
aluminum 6061 created by large strain machining. Acta Materialia, 53, 4781–4793.
doi:10.1016/j.actamat.2005.07.006
Shaw, M. C. (1986). Metal cutting principles. Clarendon: Oxford University Press.
Shekhar, S., Coi, J., Lee, S., Wang, J., & Shankar, M. R. (2009). How strains and strain-rates are accommodated
by dislocations and twins during chip formation by machining. Transactions of North American Manufacturing
Research Institute/Society of Manufacturing Engineers (NAMRI/SME), 37, 637–644.
Swaminathan, S., Shankar, M. R., Lee, S., Hwang, J., King, A. H., Kazor, R. F., ... Rao, B. C. (2005). Large strain deformation
and ultra fine grained materials by machining. Materials Science and Engineering A, 410–411, 358–363.
doi:10.1016/j.msea.2005.08.139
