Effect of Severe Plastic Deformation by High Pressure Torsion Process on Aluminium 6082 alloy

VIVEK SENGUPTA

NIT TIRUCHIRAPPALLI

Introduction

Severe plastic deformation (SPD) is one of the most promising methods for fabricating bulk ultrafine grained (UFG) materials for use in industrial applications. During the SPD process, grain refinement is achieved by imposing very large strains in the samples through plastic deformation without any concomitant changes in their cross sectional dimensions. It is well known that the plastic deformation of metallic materials depends upon dislocation slip and twinning as two of primary deformation mechanisms. Most investigations have focused on grain refinement mechanisms in metals with relatively high stacking fault energy so that dislocation slips as the dominant deformation process. For these metals, it was found that dislocation cell or low angle grain boundaries are first formed through dislocation accumulation at the beginning of the SPD process and additional deformation then transforms these interfaces into high-angle GBs through dislocation rearrangement and/or grain rotation. For metals and alloys having low values for the SFE, in which twinning may play a major and even dominant role in the plastic deformation, the grain refinement mechanism during SPD processing is not clearly defined. Nevertheless, there are some investigations showing that, when using the same SPD process, the material with a lower SFE may have a smaller grain size. The UFG materials processed by SPD generally have high strength but relatively low ductility is attributed to insufficient strain hardening due to an inability to accumulate dislocations.

SPD processing has become an attractive procedure for producing alloys with superior mechanical properties including high strength at ambient temperatures. SPD can be incorporated by processes like Equal channel angular pressing (ECAP) or High pressure torsion (HPT).

The following project deals with microstructure analysis of samples using optical and scanning electron microscope, deformed by severe plastic deformation by HPT process. And variation of hardness along the axis of the samples with different turns using Vickers micro hardness tester.

Optical Microscope

The optical microscope, often referred to as the "light microscope", is a type of microscope which uses visible light and a system of lenses to magnify images of small samples. Basic optical microscopes can be very simple, although there are many complex designs which aim to improve resolution and sample contrast. The image from an optical microscope can be captured by normal light-sensitive cameras to generate a micrograph.

Magnification

The actual power or magnification of a compound optical microscope is the product of the powers of the ocular (eyepiece) and the objective lens. The maximum normal magnifications of the ocular and objective are 10× and 100× respectively giving a final magnification of 1000×. When using a camera to capture a micrograph the effective magnification of the image must

take into account the size of the image. This is independent of whether it is on a print from a film negative or displayed digitally on a computer screen.

Scanning Electron Microscope

A scanning electron microscope (**SEM**) is a type of electron microscope that images a sample by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition, and other properties such as electrical conductivity. The types of signals produced by an SEM include secondary electrons, back-scattered electrons (BSE), characteristic X-rays, light (cathodoluminescence), specimen current and transmitted electrons. Secondary electron detectors are common in all SEMs, but it is rare that a single machine would have detectors for all possible signals. The signals result from interactions of the electron beam with atoms at or near the surface of the sample. In the most common or standard detection mode, secondary electron imaging or SEI, the SEM can produce very high-resolution images of a sample surface, revealing details less than 1 nm in size. Due to the very narrow electron beam, SEM micrographs have a large depth of field yielding a characteristic three-dimensional appearance useful for understanding the surface structure of a sample. This is exemplified by the micrograph of pollen shown to the right. A wide range of magnifications is possible, from about 10 times (about equivalent to that of a powerful hand-lens) to more than 500,000 times, about 250 times the magnification limit of the best light microscopes. Back-scattered electrons (BSE) are beam electrons that are reflected from the sample by elastic scattering. BSE are often used in analytical SEM along with the spectra made from the characteristic X-rays. Because the intensity of the BSE signal is strongly related to the atomic number (Z) of the specimen, BSE images can provide information about the distribution of different elements in the sample. For the same reason, BSE imaging can image colloidal gold immuno-labels of 5 or 10 nm diameter which would otherwise be difficult or impossible to detect in secondary electron images in biological specimens. Characteristic X-rays are emitted when the electron beam removes an inner shell electron from the sample, causing a higher energy electron to fill the shell and release energy. These characteristic X-rays are used to identify the composition and measure the abundance of elements in the sample.

Magnification

Magnification in a SEM can be controlled over a range of up to 6 orders of magnitude from about 10 to 500,000 times. Unlike optical and transmission electron microscopes, image magnification in the SEM is not a function of the power of the objective lens. SEMs may have condenser and objective lenses, but their function is to focus the beam to a spot, and not to image the specimen. Provided the electron gun can generate a beam with sufficiently small diameter, a SEM could in principle work entirely without condenser or objective lenses, although it might not be very versatile or achieve very high resolution. In a SEM, as in scanning probe microscopy, magnification results from the ratio of the dimensions of the raster on the specimen and the raster on the display device. Assuming that the display screen has a fixed size, higher magnification results from reducing the size of the raster on the

specimen, and vice versa. Magnification is therefore controlled by the current supplied to the x, y scanning coils, or the voltage supplied to the x, y deflector plates, and not by objective lens power.

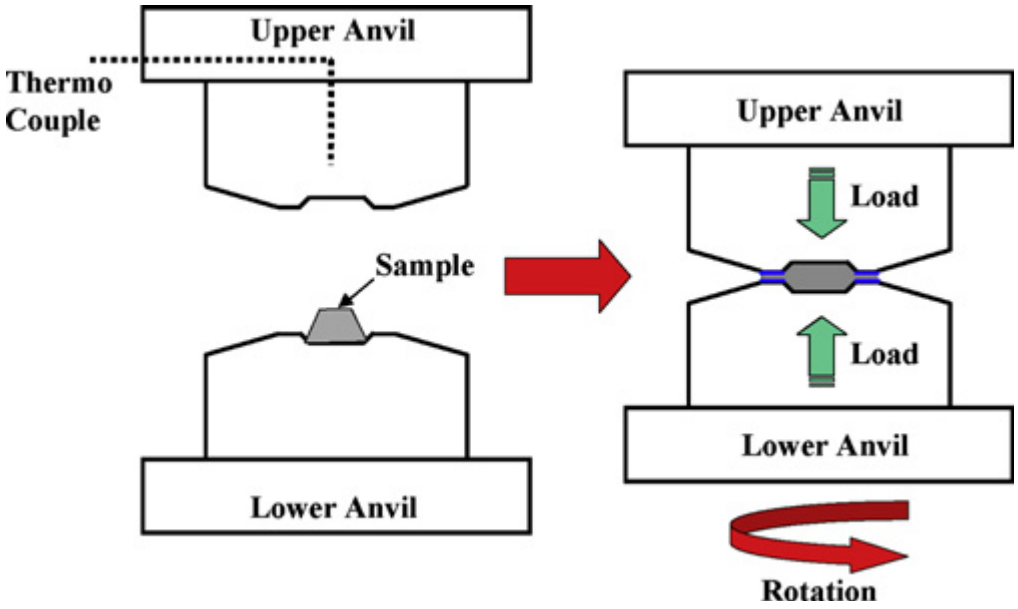


Figure 1. A schematic illustration of HPT process.

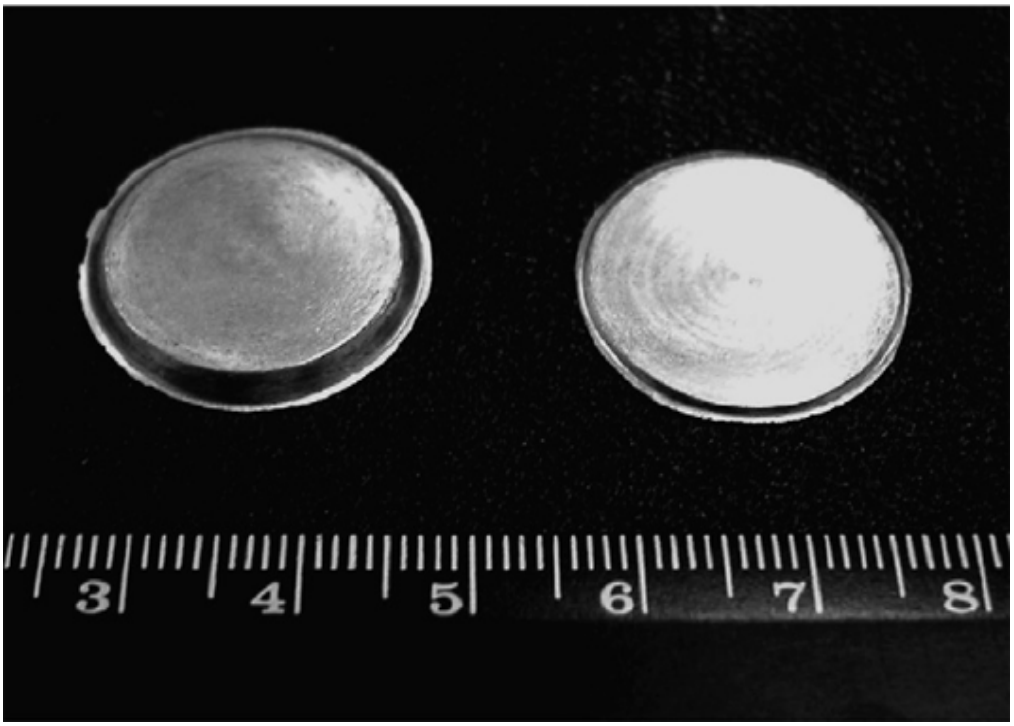


Figure 2. HPT samples (diameter 14 mm and thickness 1 mm)

Experimental procedure

A) Sample Preparation

Samples with diameters around 14 mm and thickness around 1 mm are taken for the HPT process and are severe plastically deformed under a load of around 4 GPa with different number of turns ranging from ¼, ½, ¾, 1, 3, 5 etc.

The samples thus prepared by the HPT process are mounted on mounts using black phenolic powder to prepare a back lad of the given sample. The mount in this case is non-conducting. Bainmount machine is used for this purpose. Temperature of about 180°C and heat time is about 10 minutes. Cooling time is applied of about 15 minutes at cooling temperature of about 35°C. Heater amperes is around 6.1 A and pressure is maintained around 80-100 kg cm^{-2}. After this the mounting machine is left for some time say 30 minutes for complete cooling and then the mount is removed from the machine containing the mounted samples.

Sample polishing: Sample polishing is the most important step required for the microstructural analysis of the given samples. As Aluminium is a soft sample proper care should be taken in order to polish the sample.

Firstly, check whether the sample surface is completely flat or not. This can be observed by placing the sample on a flat surface, if light comes through the bottom then the sample surface is not flat in that case go for belt grinding.

Next after grinding we go for paper polishing the sample. Start from paper number 320 if grinding is done or a start from paper number 600 should be sufficient as aluminium sample is soft. Polish using papers with numbers 600, 800, 1000, 1200, 2000 one by one such that scratches formed on every paper should be along one direction only. Also rotate the sample 90° while changing from one paper to the next.

After the paper polishing is over go for cloth polishing. Alumina powder is used. Add alumina powder dissolved in water from time to time and add water to the cloth to prevent friction thus avoiding scratch.

After alumina polishing go for silica polishing and meanwhile study the surface of the under an optical microscope so as to see any scratches present and thus try to remove them.

Finally after obtaining a scratch free surface wash the sample surface by clean water and ethanol and dry it using hot air blower. Properly wrap the sample in a tissue paper and keep it for further use.

B) Hardness measurement using Vicker's Microhardness Tester

Leica Vickers microhardness tester is used in order to calculate the hardness of the samples at different points and axes. The least count of the scales 0.01mm.

Take the prepared sample with mounting and place it between the two holders of the microhardness tester. Care should be taken that the sample is flat and not tilted as that might damage the indenter.

The indenter used is a diamond indenter and indentation surface will be square.

We are going to measure hardness on two semi axes and then will rotate the sample around 45° and again measure for two semi axes.

The hardness procedure involves the following:

Measure hardness at points along axes with 1 mm apart.

For each point take five readings, one reading in each north, south, east and west directions all 0.025 mm and fifth at the origin corresponding to these points.

The hardness of the point will thus be the average of the hardness values of these five points.

After the completion of both x and y semi axes rotate the sample by 45°, and calculate the readings on the other two semi axes.

Plot the curves with hardness on y-axis and distance on x-axis using origin.

C) Optical Microscopy

Cut the samples using diamond cutter from the centre. Miracut 125 low speed precison cutter is used and it is operated at 5-6 rpm.

Mount the cut samples and go for sample preparation.

After preparation of the sample go for etching. Etchant used is kellar's reagent having the following composition: 1% HF + 1.5% HCL + 2.5% HNO_3 rest water.

After etching go for optical analysis of the sample.

Different magnifications are used like 500x,1000x etc.

Study the microstructure and capture the images.

D) Scanning Electron Microscopy

The basic requirement for this should be that the sample should be conductive so, copper mount is used instead of Bakelite mount.

After mounting the sample is carefully polished and prepared and then etched.

Clean etched sample is fixed in the SEM and analysis of the sample is done at different magnifications like 2000x, 5500x etc.

Focus the microscope at different areas.

Pictures are taken at different points in the sample and study is done.

Result and conclusion

Hardness Analysis

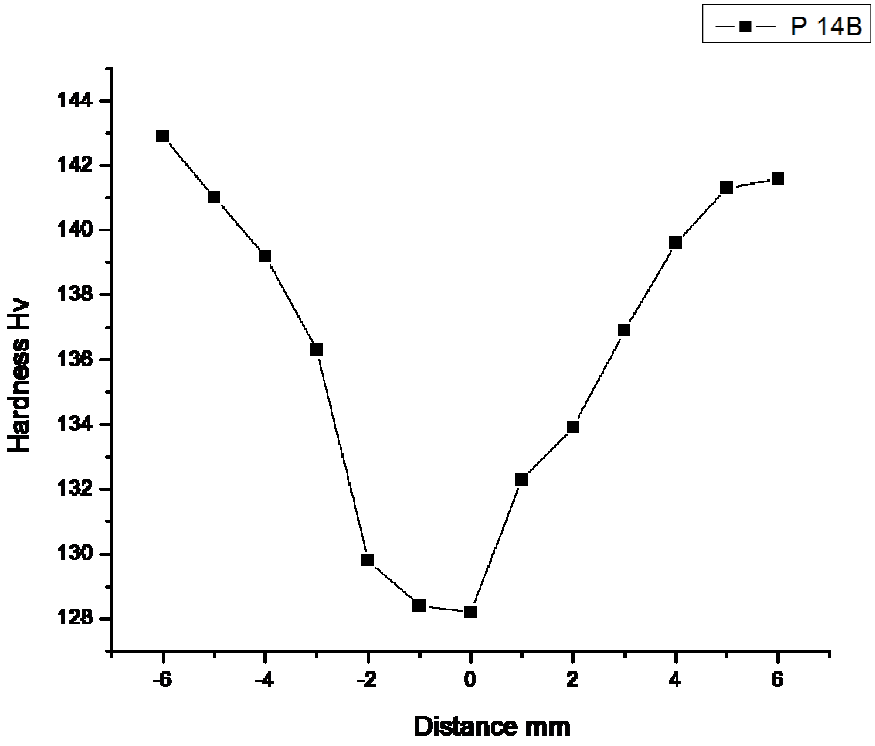
The hardness increases with the increase in number of turns as strain increases with number of turns and amount of turns. Microstructural study shows that nano-sized grains. So increase in hardness can be directly related to Hall-Petch equation.

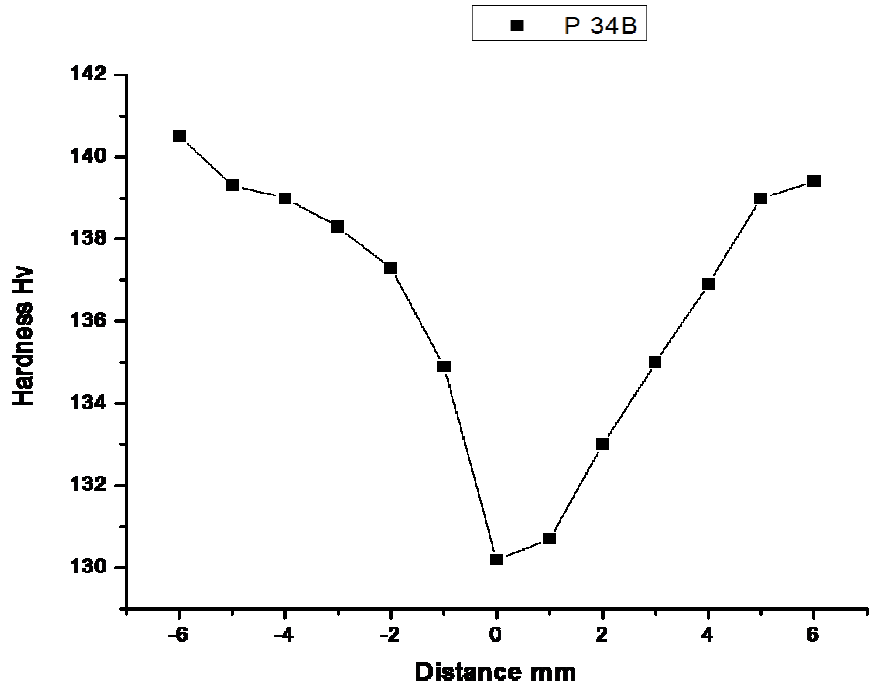
Also hardness increases from centre to edge as strain increases from centre to edge due to increase in torque with distance from centre.

The strain is well defined by

$$\gamma = r\theta/h$$

Θ is the twisting angle, h is the height of the cylindrical specimen, r is the distance from torsion axis. The dependence of γ on r is particularly helpful for our studies. The hardness vs distance curve is plotted on the next page for aged samples having ¼, ¾ and 1 turn.


P 14B
Hardness Hv
144
142
140
138
136
134
132
130
128
-6
-4
-2
0
2
4
6
Distance mm


P 34B
Hardness Hv
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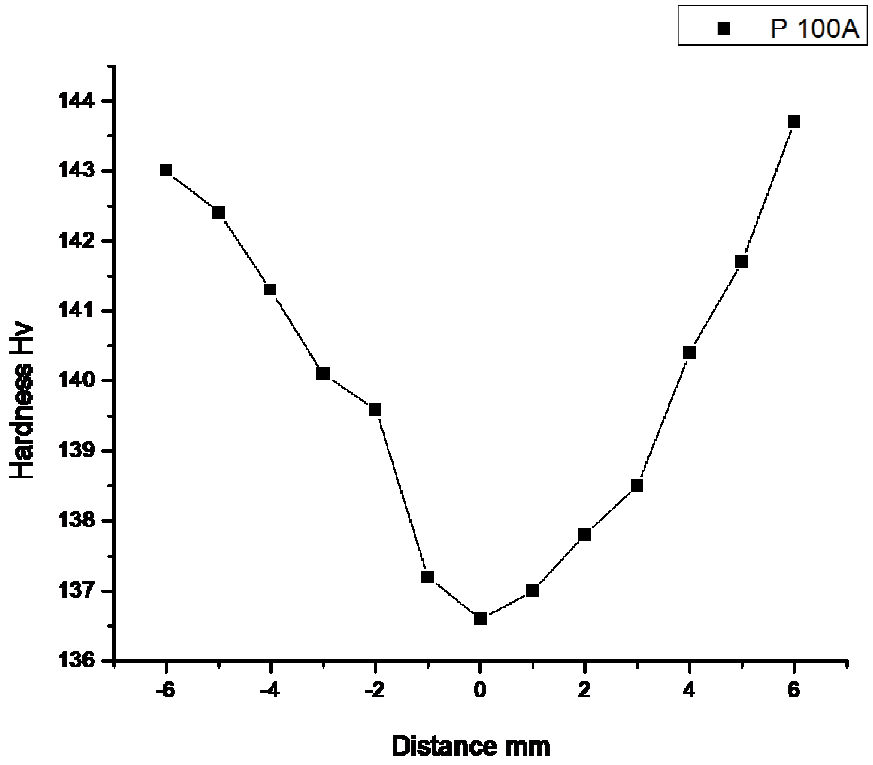
140
138
136
134
132
130
-6
-4
-2
0
2
4
6
Distance mm

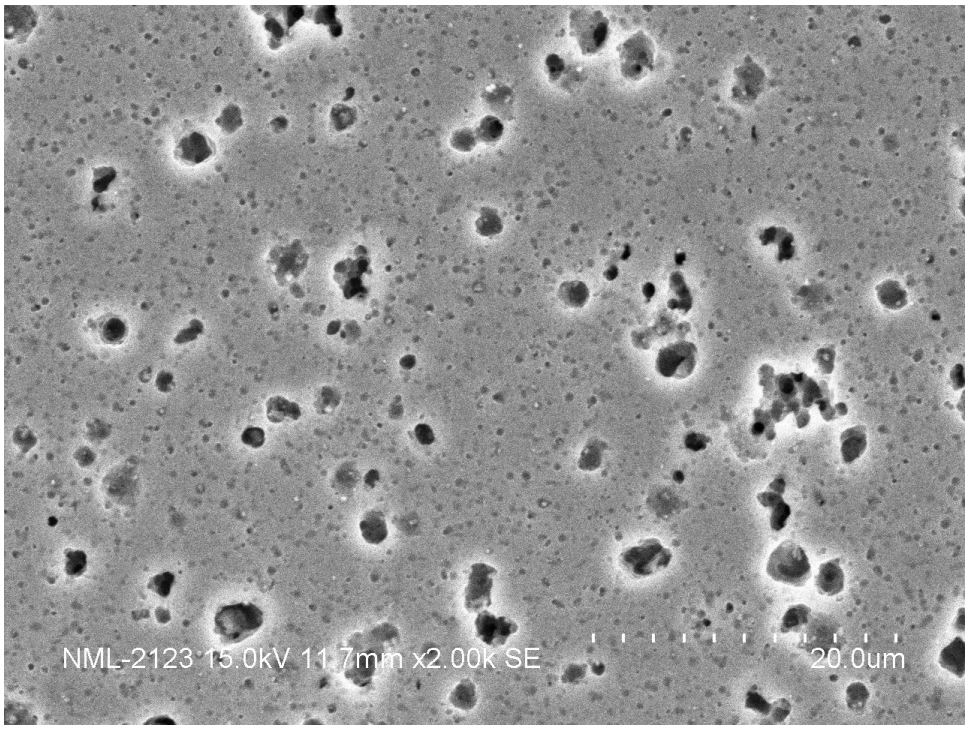


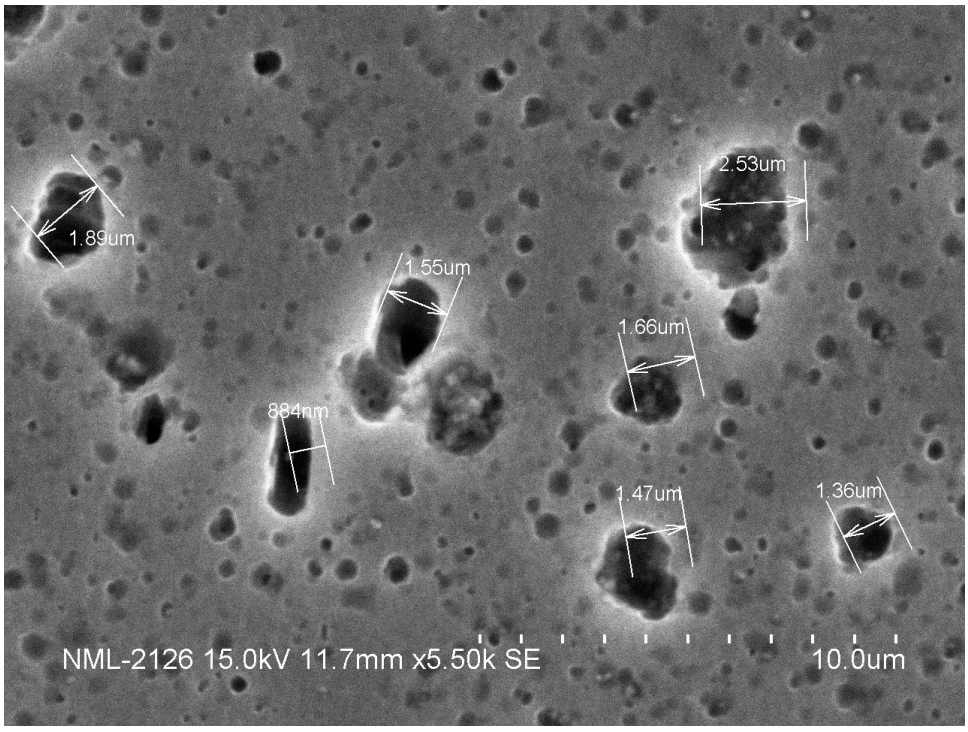
Optical microscopy

The microstructure obtained contained grains which cannot be distinguished properly as they are in sub micron or nano size. So SEM analysis is required. In aged samples the precipitates of Mg_2Si are visible as small dots.

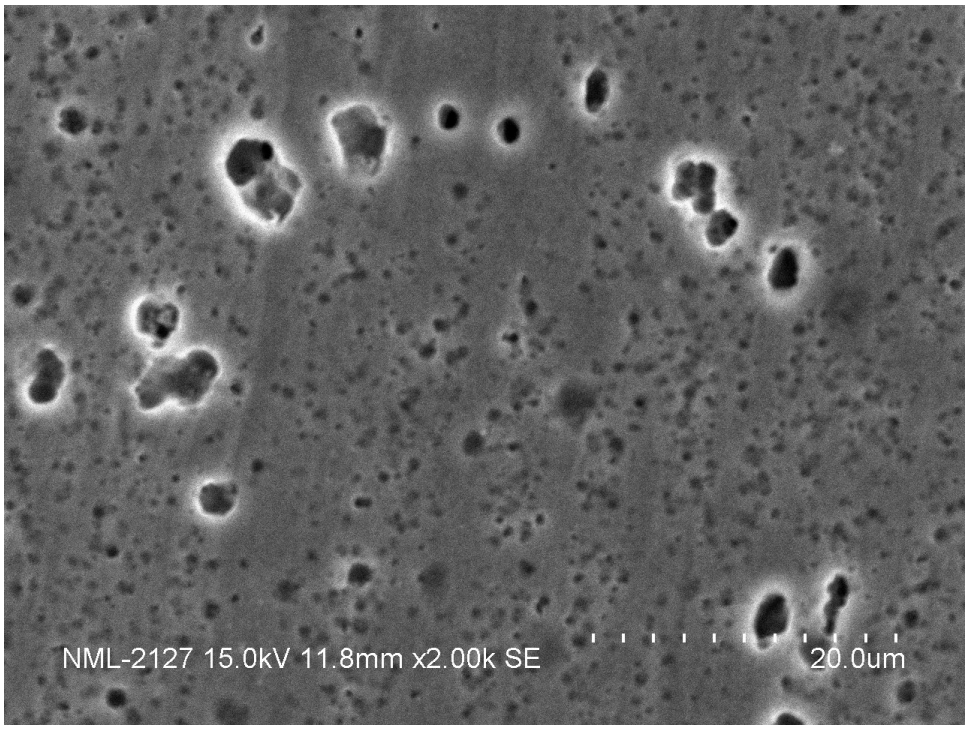
Scanning electron microscopy

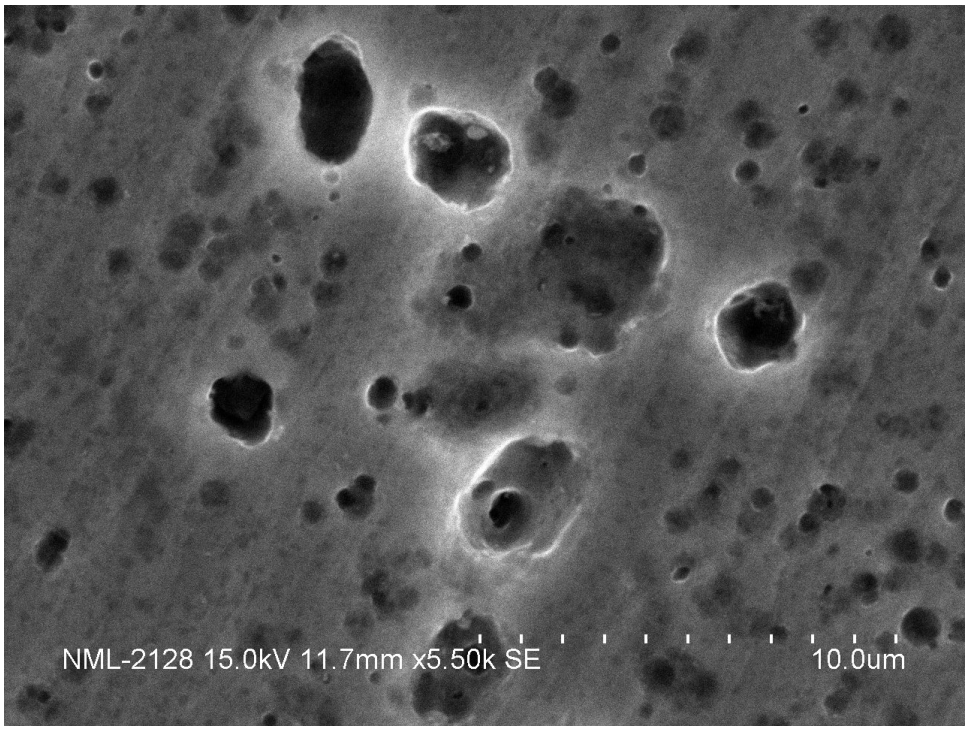
The scanning electron microscopy was done for aged samples deformed under 1,3 and 5 turns. The samples SEM showed distribution of nano sized and micron level precipitates throughout the nano size grains. The grain observed as by the SEM analysis becomes smaller as the number of turns for deformation increases. Increase in the number of precipitates is also observed with increase in number of turns. The SEM pictures of the samples is shown in the coming pages.



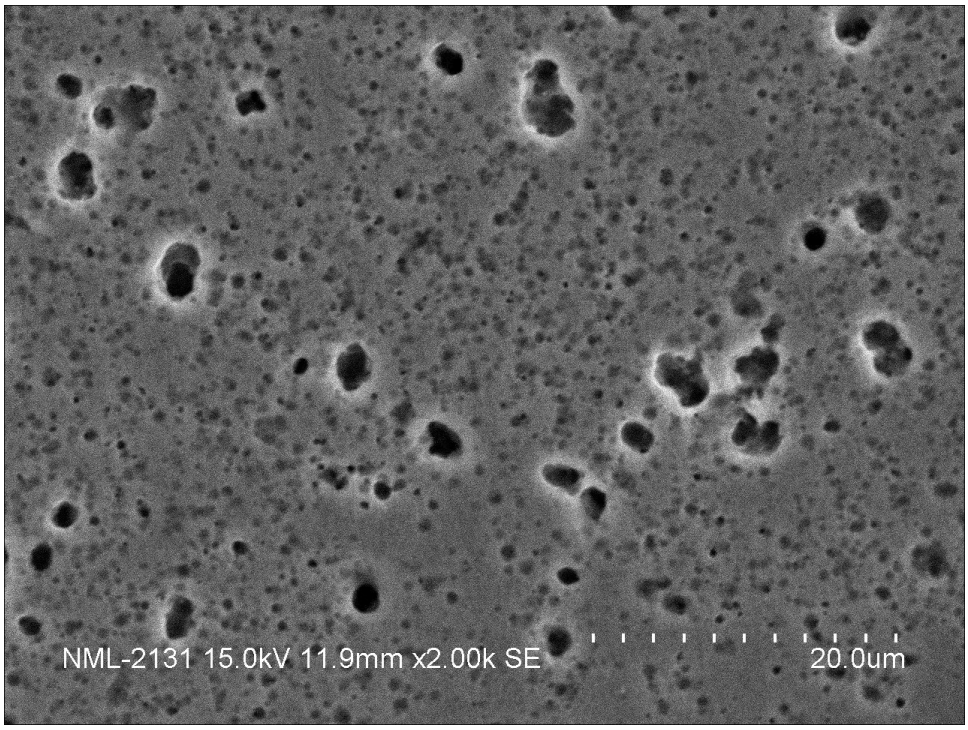


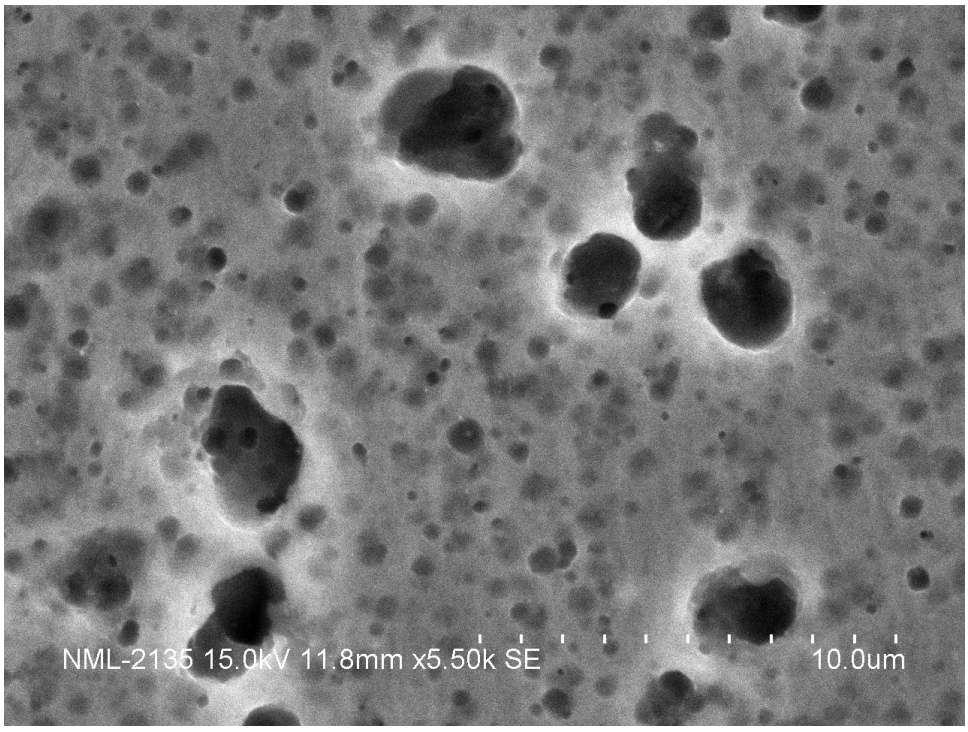
Aged sample deformed by HPT 4Gpa , N=1.





Aged sample deformed by HPT 4Gpa , N=3.





Aged sample deformed by HPT 4Gpa , N=5.