Nucleation During Recrystallisation in Ti-SULC Steel

THE UNIVERSITY of LIVERPOOL

N.C.A. Seaton and Proff D.J. Prior
Department of Earth and Ocean Sciences
University of Liverpool, 4 Brownlow Street, Liverpool L69 3GP, UK

corus EPSRC

1. Abstract

My research looks at thin sheet steel which has been cold rolled out from a large rectangular block. This leads to several things, firstly the grains become deformed, secondly a crystallographic preferred orientation is developed, and thirdly the material is work hardened due to the large amount of strain (70%) energy stored as dislocations in the grains. This third point is a problem as it means the final sheet is hard to work into useful shapes such as drinks cans and car body panels without it breaking. So the steel is annealed by heating it to around 800°C, this causes two things to happen, firstly the steel recovers by allowing some of the dislocations to move around and order themselves into a lower energy group, secondly the steel will recrystallise. It does this by nucleating new strain free grains which grow to consume the old deformed grains, this is not a random process. The new grains appear at specific sites and grow in certain ways, they also have an orientation relationship to the old deformed grains such that a crystallographic preferred orientation is developed in the new strain free material. By controlling these processes the steel can be given very good properties for forming into the new parts without breaking. My work specifically focuses on trying to understand the processes that go on during the nucleation stage of recrystallisation with an aim at improving the steels properties by further enhancing the nucleation and growth of a particular orientation.

2. Technical Abstract

The problem of texture formation during recrystallisation has been studied for a long time. It is accepted that during annealing treatments two things happen. Firstly recovery takes place and this is then followed by nucleation and growth of new strain free grains. This leads to the final texture of the material being derived from both nucleation and subsequent growth. Humphreys and Hatherly [1] make two main points: the nuclei already pre-exist in the volume and that during recrystallisation nucleation is nothing more than sub grain growth that occurs heterogeneously. More recently this problem has been looked at in relation to interstitial free (IF) steels of which Ti stabilised ultra low carbon (Ti-SULC) steel is one. These have good formability because of the texture developed and some common conclusions can be drawn [2-4]:
1) Recrystallisation starts with nucleation in areas of high strain such as grain boundaries and intragrain shear bands in {111}<uvw> type grains.
2) Grains of the {hkl}<110> type recrystallise at a later stage due to their relatively low stored energy.
3) There are no preferential misorientation axes for migrating recrystallisation fronts.
4) Sluggish and incomplete recrystallisation may be due to reduced stored energies through recovery processes in the non-recrystallised areas.
The one limiting factor of these studies is that they look at fixed stages during recrystallisation by quenching the sample and then analysing it. But to understand fully a dynamic process such as this you must observe it directly whilst it is happening. This problem was recently overcome with the development of in-situ heating experiments inside a SEM to allow EBSD data to be gathered during the experiments [5]. This technique allows heating of the sample with fully automated EBSD analysis at up to 50 points per second so that observed microstructural changes can be directly linked to measured crystallographic data thus allowing interpretation of the kinetics.

3. Experimental techniques

Samples were cut with a maximum size of 9 x 9 x 1 mm. These were prepared for EBSD analysis. Experiments were performed in the CamScan X500 crystal probe field emission gun SEM. This satisfies the EBSD requirement of a high angle of tilt between the sample and the electron beam by tilting the electron column over so the sample can remain horizontal for heating experiments (Fig 1a). The hot stage [5] itself consists of a brass body with a water cooled plate; inside this is a carbon block around which are the heaters. On top of block there is a small hole where the sample sits; spacers are used to bring the specimen surface up level with the stage's top surface to optimise the visible area for OC imaging (Fig 1b).

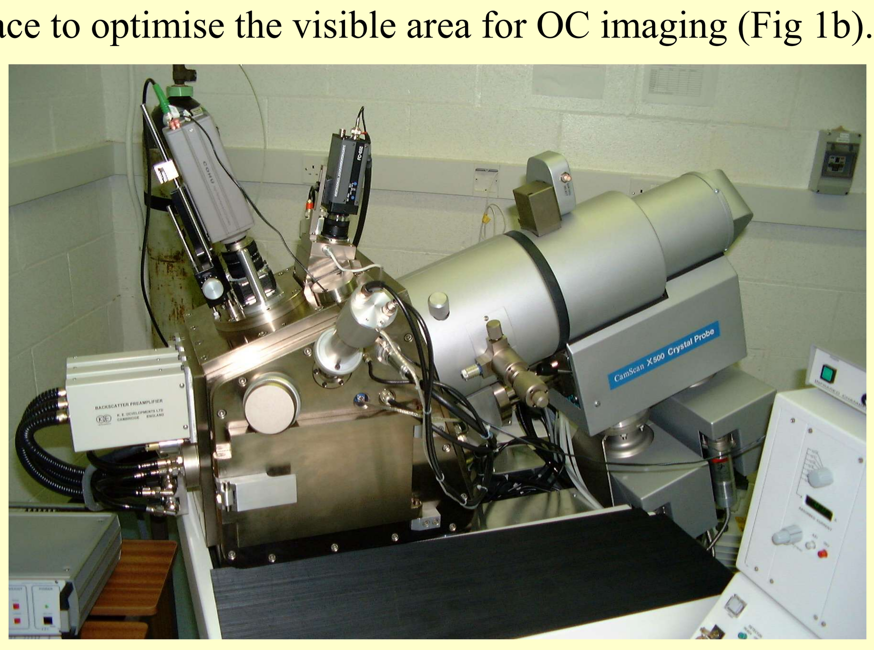
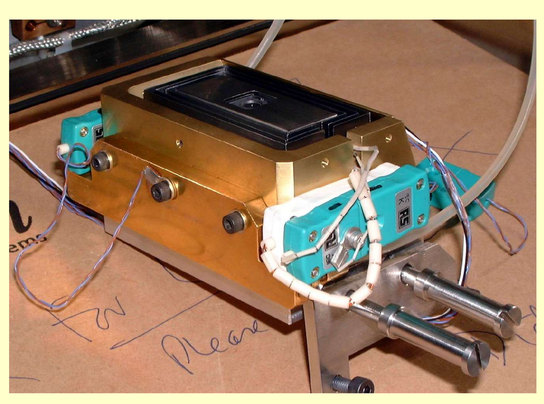
(a)  (b) 

Figure 1(a) External view of the X500 crystal probe SEM, (b) view of the heating stage outside of the SEM

This setup allows us to take both still pictures in the back scattered or secondary electron mode and quantitative EBSD data whilst at temperature. So samples can be watched as they recrystallise and data gathered as to the crystallography of the grains that are growing or being replaced.

4. Results 1

Fig 2 shows a set of three EBSD maps taken at different stages of a heating experiment and showing the deformed structure, 50% recrystallised and 95% recrystallised, a, b and c, respectively. It can be seen that grains of the{111} type are replaced first and that the {001} grains remain longer, one still being present at the end.

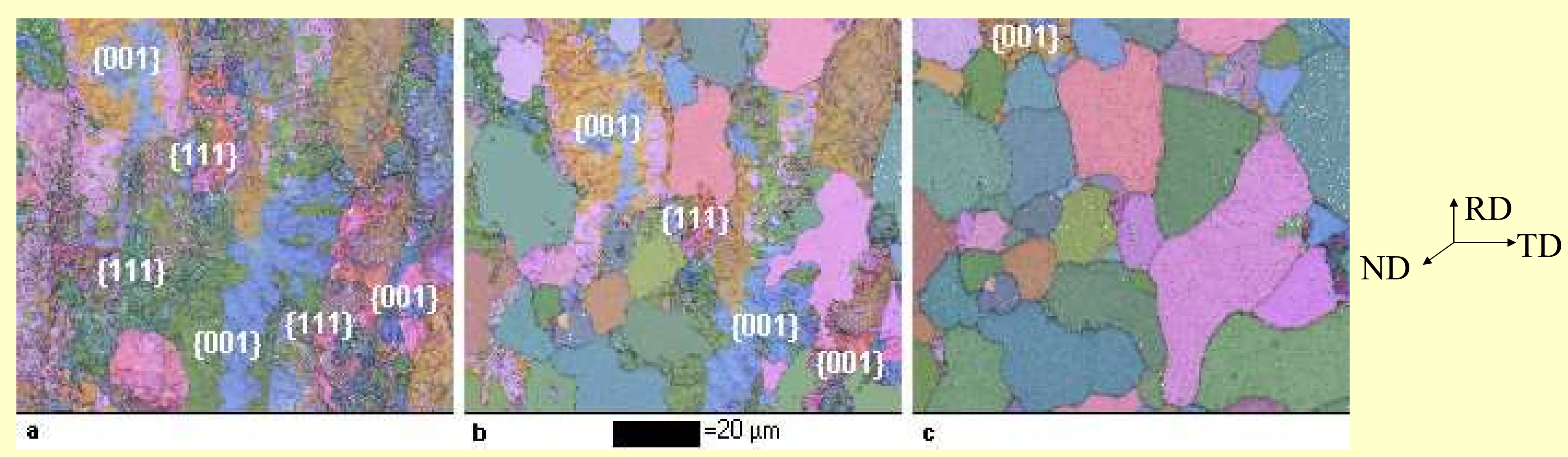
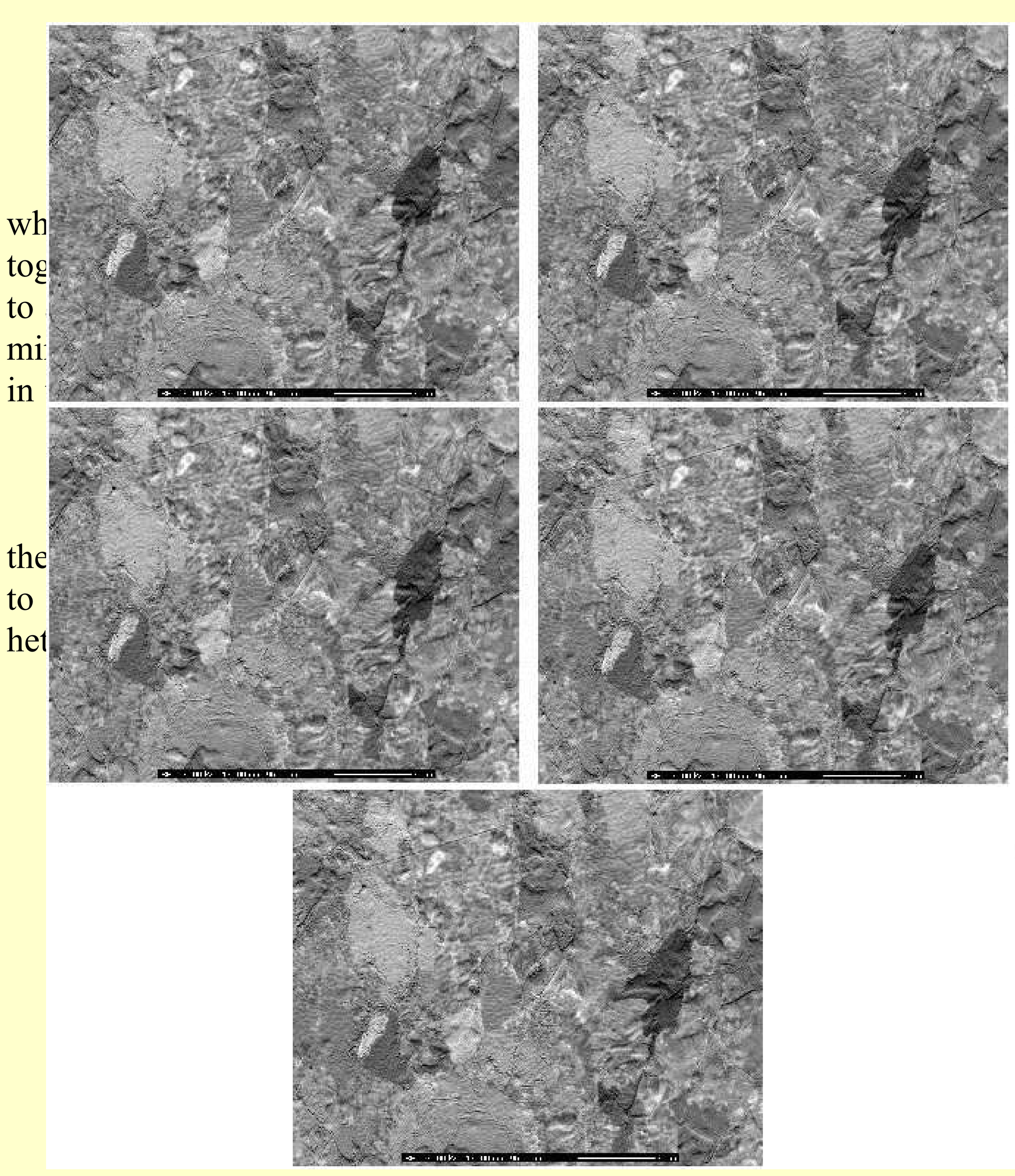


Figure 2. EBSD maps of a heating experiment showing from left, deformed, 50% recrystallised and 95% recrystallised.

The new grains appear at the boundaries of {111} grains and {001} grains and grow to consume the {111} grains. The new grains are mostly {111} type with some {001}, if the mechanism was strain induced boundary migration (SIBM) then you would expect the {001} grains to simply grow and consume the {111} grains but this is not observed.



Here in figure 3 we can see some ...ntation contrast images taken ...d b of figure 2. ... series wh... once they are put tog... frame takes three seconds to ... and there is a gap of about 2 mi... between each of the frames in ... series. This gives us the ability to ...rve the growing grains and see ... move and how they ... with the ... from the EBSD maps to ... is a reason for the het... nature of the growth.

Figure 3. Orientation contrast ...n between maps a and b

5. Results 2

Experiments on sections through the sheet in the rolling direction reveal that the {111} type grains contain many shear bands from the rolling. These can be seen as the colour changes in the grains in fig. 4, they always form in the plane defined by the rolling direction and the normal direction at 45° to the rolling direction.

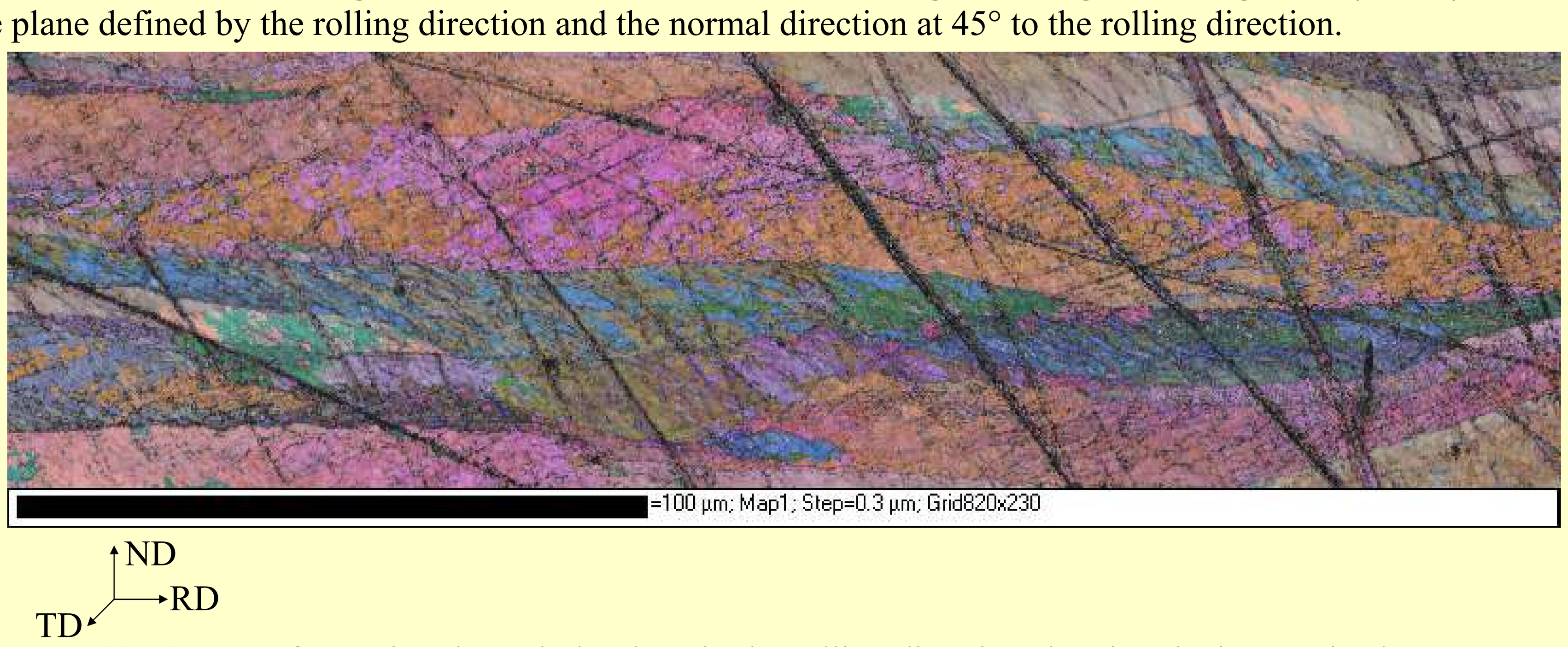


Figure 4. EBSD map of a section through the sheet in the rolling direction showing the intragrain shear banding.

6. Conclusions

Recrystallisation of Ti-SULC steel is a very heterogeneous process in both position of new grains and in their growth. However we can say that;

- {111} grains with HAGBs nucleate on the grain boundaries of deformed {111} grains.
- Nucleation and growth of {111} grains occurs at the expense of deformed {111} grains.
- {001} grains remain for longer during the anneal than {111} grains because of their relatively low stored energy.
- {001} grains tend to be consumed by new {111} grains rather than nucleate new {001} grains
- There was little evidence of growth of {001} grains into {111} grains as might have been expected from the SIBM mechanism.

References
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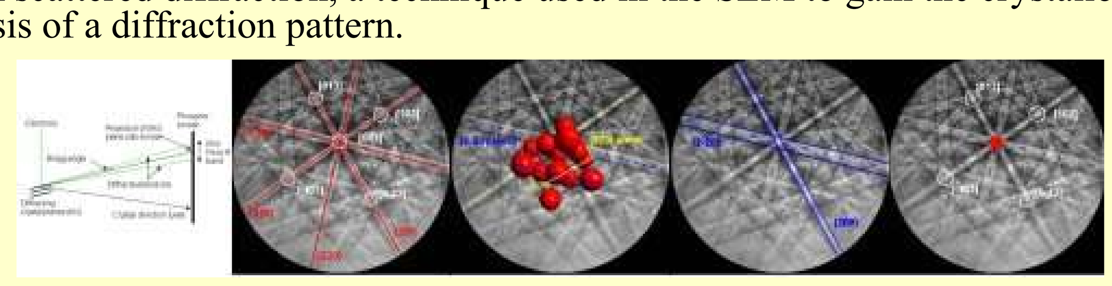
[4] I. Samajdar, B. Verlinden, P. Van Houtte and D. Vanderschueren, Scripta Mat. 37 (1997) 869-874.
[5] G.G.E. Seward, D.J. Prior, J. Wheeler, S. Celotto, D.J.M. Halliday, R.S. Paden and M.R. Tye, Scanning, 24 (2002) 232-240.

7. Glossary

SEM - Scanning electron microscope, uses electrons instead of light to observe a sample due to their smaller wavelength, see figure 1a.

EBSD – Electron back scattered diffraction, a technique used in the SEM to gain the crystallographic axis of a point by analysis of a diffraction pattern.



Ti-SULC – Titanium stabilised ultra low carbon steel. Contains 0.002 weight % carbon, 0.04 weight % Ti.

RD/ND/TD – Rolling normal or transverse direction. Reference frame of samples to cold work done to them, see figure 2 & 4.

Cold rolling – Work done to change the shape of sample, used to turn cast blocks into thin sheet. Done below the recrystallisation temperature <300°C