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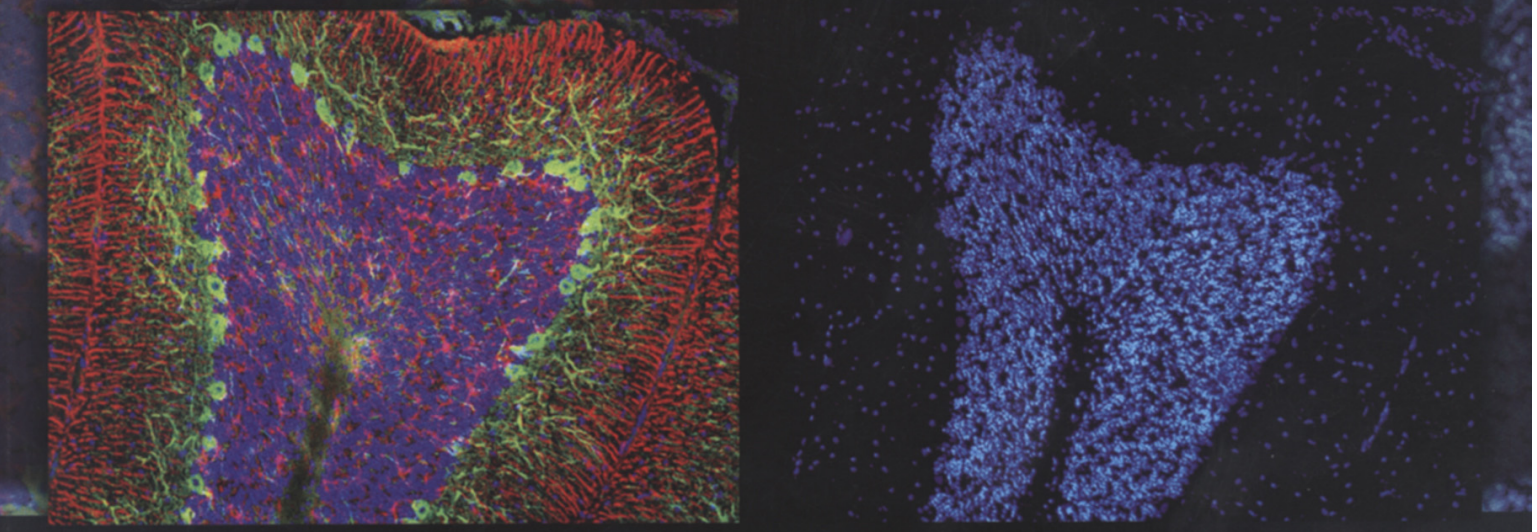

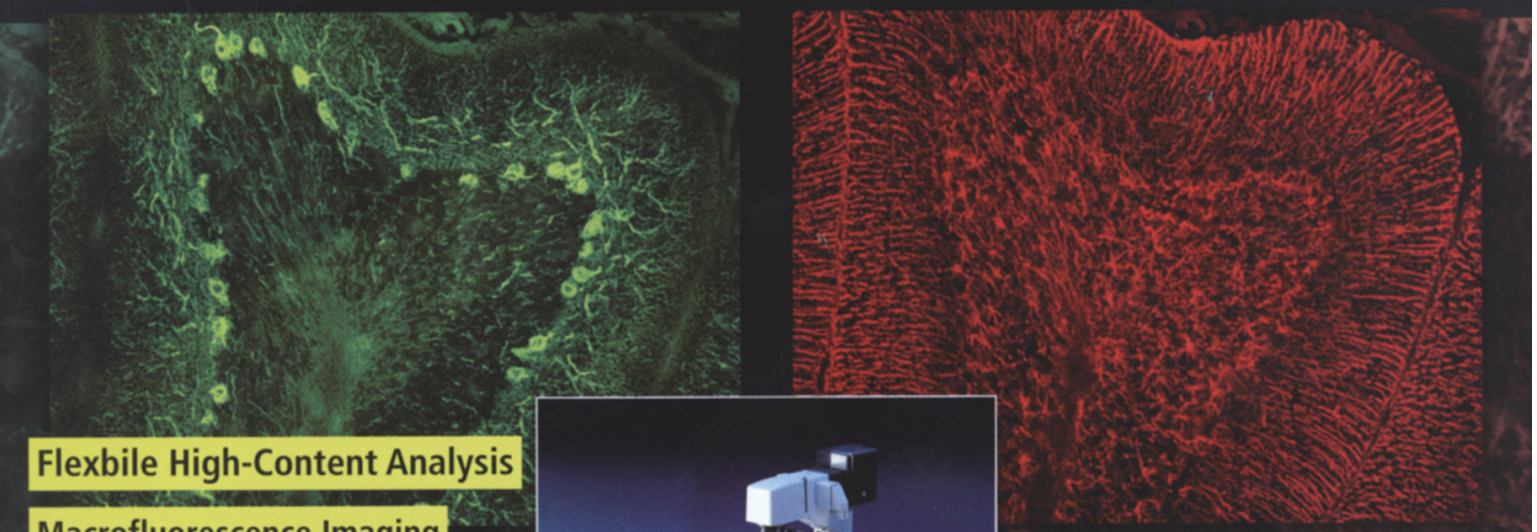

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Electron Backscatter Diffraction in a FEGSEM

A scanning electron microscope equipped with an electron backscatter diffraction system enables the quantitative analyses of the microstructure of crystalline materials, even phase analysis at individual grains is possible. This provides a better comprehension of the mechanical properties of materials and facilitates the development of materials with special predefined properties.

Introduction

Most materials are polycrystalline and especially their mechanical properties are strongly influenced by characteristics like the average grain size, the grain size distribution, textures and the crystal structure. To tune the properties of metals and alloys with respect to these parameters, recrystallisation of the respective materials at specified temperatures, strain rates and strains is simulated.

Traditionally, the size distribution of the grains and the recrystallised fraction were determined with optical microscopy, whereas the crystallographic texture was measured with x-ray or neutron diffraction. The advantage of these techniques is the possibility to measure large volumes of a specimen within a rather short time. In the opposite, electron backscatter diffraction (EBSD) is a relatively time-consuming analysis method, but has the great advantage that data (e.g. grain size, orientation, disorientation between grains, phase information) are gained from single grains, which can be used to elucidate direct neighbourhood relationships between these individual grains [1]. With a field emission scanning electron microscope (FEGSEM), it is possible to carry out quantitative analyses of grains/subgrains as small as ~100 nm, of course depending on the type of material.

Materials, Instrumentation

The nickel base alloy 80 A was used to exhibit the capability of the EBSD technique for the determination of the recrystallised fraction. Cylindrical specimens (h=12 mm, d=10 mm) of this alloy were annealed at a temperature of 1220° C for 60 seconds, subsequently compressed with a Gleeble 3800 testing system at a temperature of 1120° C to different strains (strain rate 0.1 s^{-1}), and immediately quenched to room temperature. Transversal cross sections at a quarter of the height of the specimens were cut out for the microstructural investigation. In order to obtain EBSD patterns with high quality, the specimens were polished with an alkaline colloidal silica solution (0.04 µm granularity) as the final polishing step.

The application of EBSD for phase identification is demonstrated at a cross section of a commercially available duplex steel 1.4462 (no further treatments). The final polishing step was also performed with colloidal silica.

All the measurements and analyses were performed on a Zeiss Gemini 982 DSM (primary electron energy: 20 keV; probe current: 2.8 nA), equipped with an EDAX-TSL system (SIT camera, OIM 4 software, see figure 1). Grain boundaries were characterised by a disorientation larger than five between neighbouring measurement points.

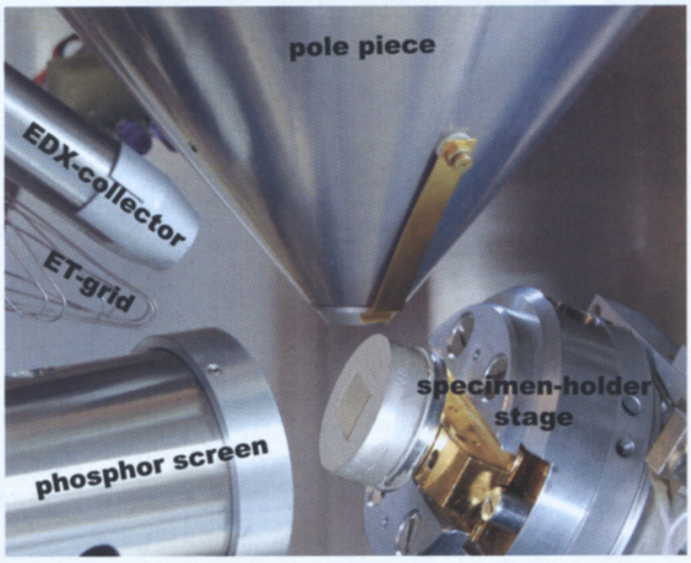

Fig. 1: Image of the SEM sample chamber with the phosphor screen for EBSD analyses inserted.

Recrystallised Fraction

Because the original and recrystallised grains differ in their deformation, size and orientation distribution, the mechanical properties of a material also depend on the fraction of recrystallised grains. The discrimination between the deformed and recrystallised grains with EBSD can be performed by several methods, but the grain orientation spread was proven to be most successful

for this nickel alloy [2]. A typical example of the grain orientation spread of a partly recrystallised specimen (strain 0.5) and a fully recrystallised specimen can be found in figure 2. The comparison of both distributions clearly shows that the first peak of the grain orientation spread belongs predominantly to the recrystallised fraction. The deformed and recrystallised fraction can be separated by setting a threshold of the grain orientation spread at a value of 1.5° as marked in figure 2. The evolution of the resultant structures is illustrated in figure 3 and is represented there as inverse pole figure map (IPF). From this figure it can be seen that the recrystallisation predominantly starts at the edges of grains and mainly takes place at 'normal' grain boundaries and not at twin boundaries. Additionally, the recrystallised grains form closed networks very soon, which result in the well-known necklace structure. Since the IPF shows a statistical distribution of the orientation of the individual recrystallised grains, definitely no texture is present. Additionally, figure 3 depicts the much smaller grain size of the recrystallised grains compared to the original ones. Furthermore, a nearly linear correlation between the strain of the specimen and the amount of recrystallised grains was found.

Ferrite – Austenite

An important parameter for the properties of a duplex steel is its ferrite fraction. With EBSD it is also possible to determine the phase information of a material down to a micrometer scale (e.g. [3]). An important prerequisite for a successful differentiation between two phases is that they differ either significantly in their lattice parameters or in their crystal structure. The small wavelength of the electrons entails a small Bragg angle (~ 0.5°) and thus makes exact calculations of lattice plane spacing rather difficult. In the case of ferrite and austenite, a cubic body centred and face centred crystal system exists. As a consequence, these two phases can be separated by EBSD. In figure 4a the phase map of the investigated duplex steel is shown. Since the phase of every measured point is obtained directly from the associated EBSD pattern and stored in the data set, the respective fractions can be determined without setting any threshold as it is necessary, for instance, when using light microscopy. The ferrite fraction was determined to an amount of 54 % for the investigated duplex steel. Additionally, the orientation of the grains of both phases can be displayed separately (see IPF of fig. 4b and c). From these figures it is obvious that the austenite phase contains a lot of twinned grains, which is a general phenomenon for face centred cubic materials. Nearly no twinning can be observed in the ferrite grains.

Summary

The investigations clearly demonstrate the capability of EBSD in conjunction with a FEGSEM for the quantitative analysis of the structure of crystalline materials. The opportunity to gain direct neighbourhood relationships between individual grains enables a better understanding of the influence of the manufacturing process on the microstructure and in addition on the macroscopic properties of the materials. This knowledge is the prerequisite for the development of new materials.

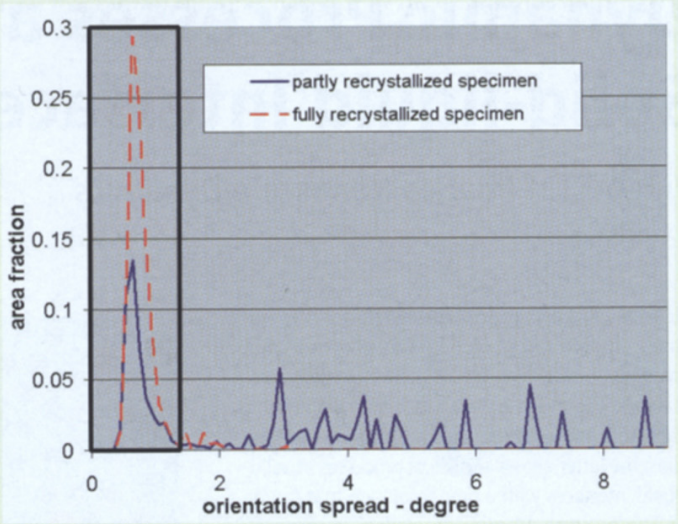

Fig. 2: Grain orientation spread of a partly recrystallised specimen (blue, strain 0.5) and a fully recrystallised specimen (red) with the range for the determination of the recrystallised fraction market.

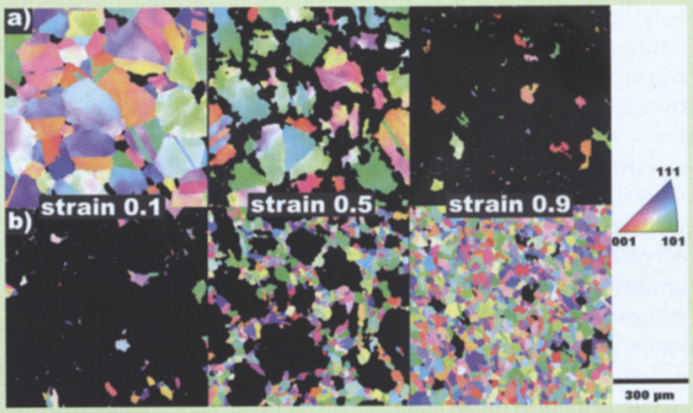

Fig. 3: Inverse pole figure maps of a) the deformed and b) the recrystallised grains of nickel alloy specimens treated with different strains.

Fig. 4: a) phase map (blue ferrite, red austenite), b) IPF of the ferrite phase and c) IPF of the austenite phase of the duplex steel

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