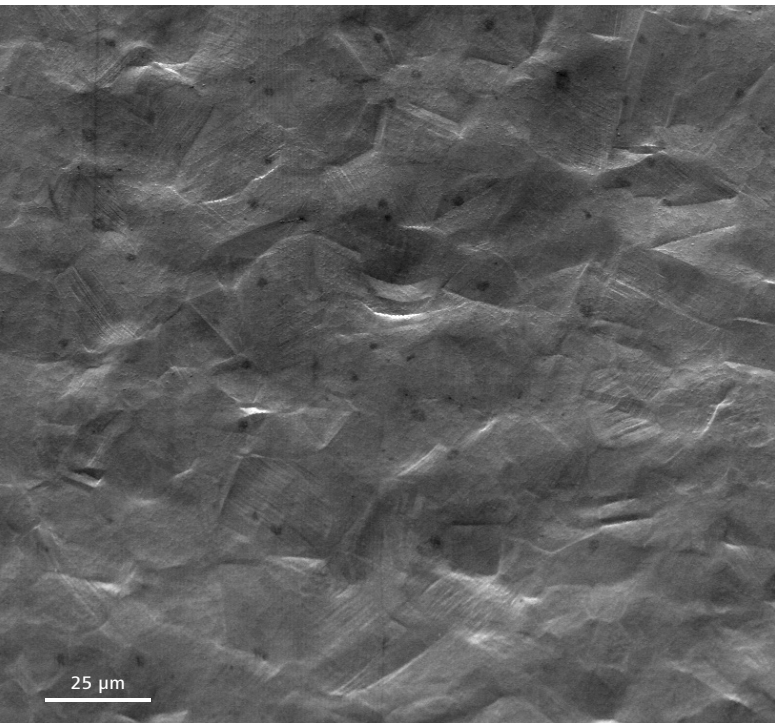
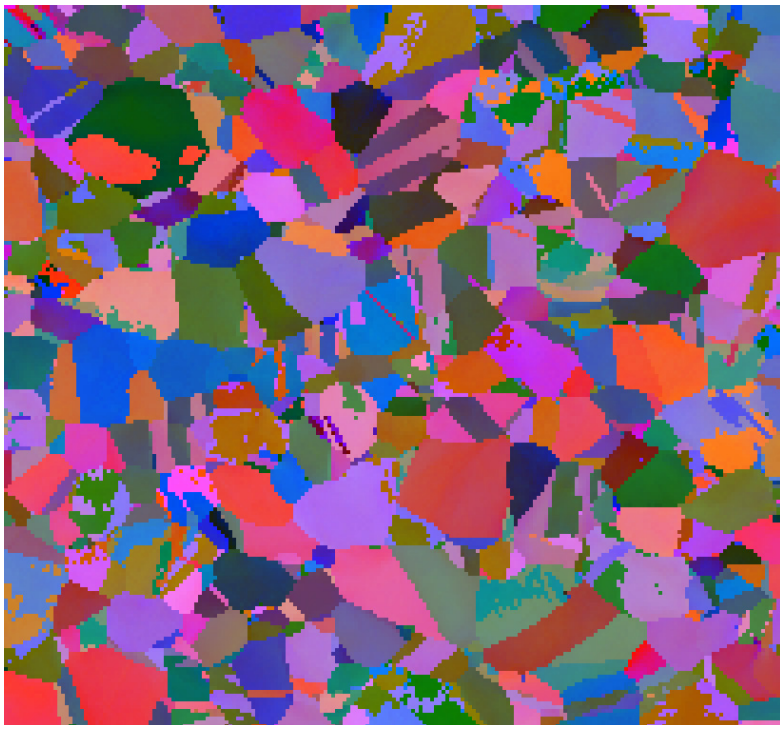
White Paper

Revealing Plasticity Progression in Steels via Observation of *in situ* EBSD Deformation

In Situ Lab for ZEISS FE-SEM





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Seeing beyond

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Tensile testing is essential for the mechanical characterization of metals. Being able to combine mechanical testing with imaging modalities offers unique insights into the deformation behavior of engineering materials since the deformation progression can be tracked by observation. In this work, the *In Situ* Lab for ZEISS FE-SEMs (field emission scanning electron microscopes) is used to perform tensile tests on steel samples at high temperatures while tracking the progression of the deformation using SE (secondary electron) and EBSD (electron backscatter diffraction) imaging. Through correlation of the SE and EBSD data, high Schmid factor grains can be identified as main contributors to the deformation within the microstructure. Their crystallographic orientation changes throughout the deformation progress, which is tracked in EBSD contrast. This is a result of a series of dislocations moving through the grains, each leading to a slight misorientation. This effect can be tracked and localized using the given test setup. Further SE imaging reveals the morphological change at the sample surface, as plasticity progresses in the bulk of the material.

Introduction

In materials science, tensile testing is the backbone of mechanical characterization of metals. The ability to combine mechanical testing with advanced imaging and characterization methods and the option to operate at high temperatures up to 800 °C opens a large variety of possibilities. Different annealing states can be achieved and tested at high temperature or after cooling to room temperature. Using the EBSD information, high Schmid factor grains can easily be identified and monitored during the *in situ* tensile experiment and therefore even the first yielding grains are captured. Further, the chemical information of the sample can be collected simultaneously via EDS (energy dispersive X-ray spectroscopy). Even BSD (back scatter diffraction) contrast imaging throughout the whole deformation process can be used to achieve ECCI (electron channeling contrast imaging) conditions. By using feature tracking, the chosen region of interest remains in the field of view and is imaged throughout the whole deformation process, enabling full automation of the experiment. Such experimental capability paves the way for high throughput data collection to build up a database of microstructural characteristics of the investigated material in combination with macroscale material performance.

Materials and Methods

The tensile specimens were prepared as dog bone shaped specimens cut from a steel (X5CrNi18-10) sheet material via water jet cutting. The as-cut samples showed a thickness of 2 mm, a gauge length of 10 mm and gauge width of 2 mm, resulting in an initial cross-sectional area of 4 mm². The sample surface was prepared by grinding and polishing with a surface finish using a colloidal silica suspension with a grain size of 0.25 µm. The tests were performed using the *In Situ* Lab for ZEISS Sigma 500 VP equipped with an Oxford Symmetry S2 EBSD camera. Imaging conditions of 20 kV acceleration voltage and a beam current of 5 nA resulted in a very high index rate in the EBSD signal. The high working distance of ~20 mm was achieved by tilting the EBSD camera towards the steel sample. The steel samples were mounted in 70° tilt orientation inside the *In Situ* Lab and tested at different temperatures (room temperature, 400 °C and 600 °C). The tensile test was performed in extension control with deformation steps of 5 µm and a jaw speed of 10 µm/s. After each deformation step, the motors stopped and feature tracking and imaging were executed. EBSD imaging was performed after each sixth deformation step since the EBSD scan is usually more time consuming. The test was performed until the sample failed or the maximum force of the system was reached.

Results and Discussion

From the force and displacement data, stress and strain data were processed for the three tests at different temperatures. Evaluation of the Youngs modulus from the elastic region of each respective graph was performed, and the values were used to calculate the plastic strain by removing the elastic strain from the overall strain data. Calculating the plastic strain enabled comparison of representative strain values between experiments with different yield strengths. The stress-strain data is plotted in Figure 1.

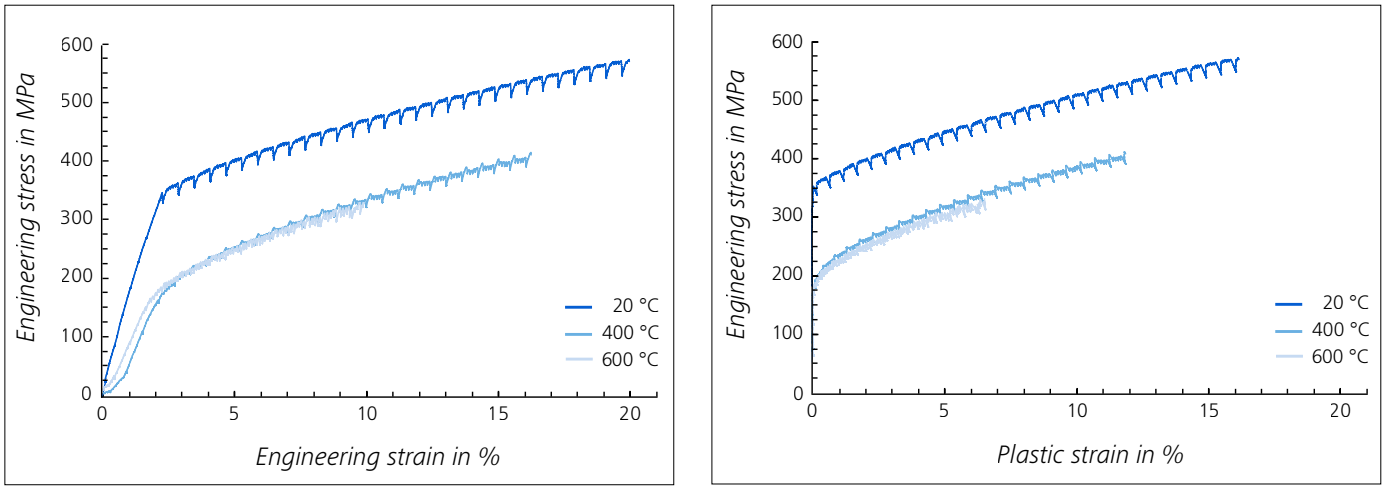


Figure 1: *Stress-strain data of X5CrNi18-10 steel at different temperatures; stress plotted vs. strain (left) and stress plotted vs. plastic strain (right).*

The mechanical data shows a reduction in yield strength at elevated temperatures (400 °C and 600 °C) compared to the measurement at room temperature. The yield strength of both tests at high temperatures is about the same and does not decrease further by increasing the temperature from 400 °C to 600 °C. All three tests show a significant strain hardening behavior throughout the measurement as the necessary stress for further deformation after yielding increases. All tests show load drops throughout the test profile, which can be attributed to stress relaxation during EBSD imaging, as the motors stop for the imaging sequence. The test at room temperature was aborted after reaching a force >2 kN, since, in the given configuration, the stiffness of the tested sample is too high compared to the stiffness of the mechanical rig. For the test, the high load bar was removed to allow the EBSD detector to come closer to the sample for optimized EBSD acquisition. By removing the high load bar, the machine stiffness was reduced, leading to instabilities while applying high loads (>2 kN). In order to fully deform the given samples to achieve final failure, a reduction in the cross-sectional area would be necessary. These instabilities are increased at elevated temperatures, which led to abortion of the tests at lower force values. Nevertheless, the strain hardening behavior of all three tests was very precisely documented for the given measurements. Further, the strain hardening revealed in the mechanical data can be correlated to the microstructural change observed in EBSD imaging. This enabled a quantification of plasticity throughout the different temperatures at a given representative plastic strain. In Figure 2, the grain orientation from EBSD imaging is shown in Euler contrast at a plastic strain of 5 % for all three measurements.

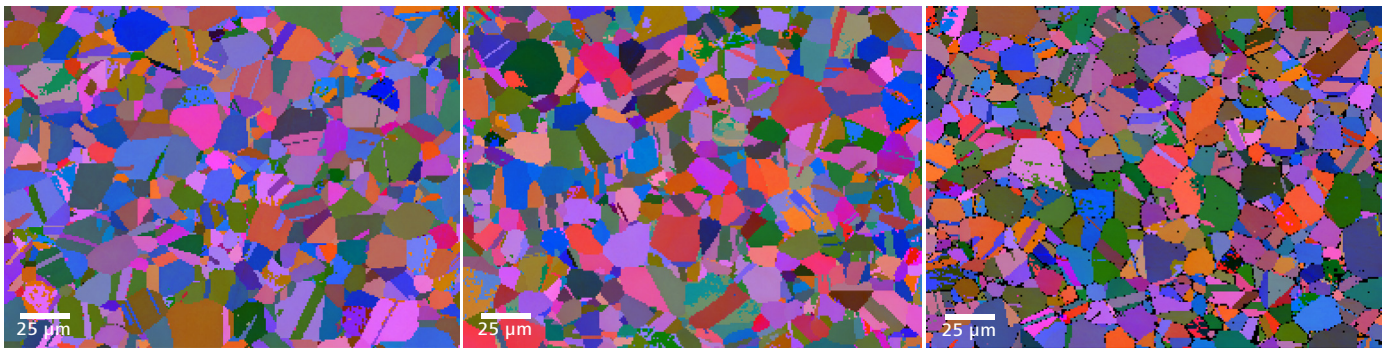


Figure 2: *EBSD maps of the microstructures in Euler contrast for the measurements at room temperature (left), 400 °C (center) and 600 °C (right) at a representative plastic strain of 5 %.*

It is apparent that specific grains within the microstructure of all three measurements already showed a misorientation. This is a result of a series of dislocations travelling through the grains, each one leading to a slight misorientation. This misorientation was captured in the EBSD data and reveals local hotspots of plasticity. Using this method, high Schmid factor grains can easily be identified as the main contributors of plasticity. Since the EBSD data was acquired throughout the whole deformation process, this comparison can be made throughout the whole plastic regime while having only performed three measurements. By using conventional tensile testing with pre- and post-deformation characterization, one measurement for each temperature would be necessary to map the stress-strain curve and reveal the overall strain for achieving a specific plastic strain at the given temperature. Then, to be able to compare the deformation behavior, one measurement would be necessary up to a specific plastic strain value. If a comparison of three plastic strain values is desired, a total of 12 experiments would be necessary in conventional tensile testing, whereas using *in situ* tensile testing only requires three measurements.

In Figure 3, a series of EBSD maps is shown for plastic strain values of 0,5 and 10 % at a temperature of 400 °C. Here, the appearance of plasticity within the microstructure can be closely followed. With increasing plastic strain, the misorientation increases within single grains throughout the microstructure. This can be observed by following the grains, marked by arrows, as their color keeps changing at higher plastic strains. This change in orientation is apparent for multiple grains in the microstructure and develops due to an increasing number of dislocations moving through the microstructure. At higher plastic strain and higher stress due to strain hardening, more and more grains with lower Schmid factors are activated, resulting in plastic deformation. Additional EBSD data is shown in the appendix. Since the data was taken inside the SEM, it was possible to establish a correlation between the EBSD maps and SEM images. A series of SEM images for the test at 400 °C and plastic strain values of 0,5 and 10 % is shown in Figure 4.

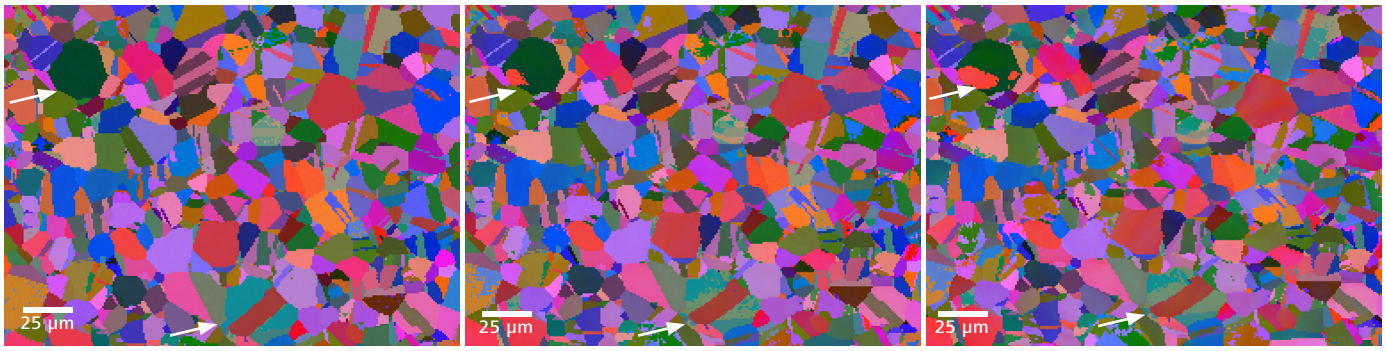


Figure 3: *EBSD maps of the microstructures in Euler contrast for the measurement at 400 °C at different levels of plastic strain 0 % (left), 5 % (center) and 10 % (right).*

As the deformation progresses, a surface morphology evolves, representing the individual grains as they tilt in different directions throughout the deformation process. This surface morphology can cause the EBSD acquisition quality to decay with progressing deformation compared to the pristine state. By acquiring EBSD and SEM information, a correlation between surface morphology and grain orientation can be established. Performing this *in situ* investigation enables identification of strain localization at high Schmid factor grains and the development of plasticity of these early-to-deform grains while observing the surface morphology development.

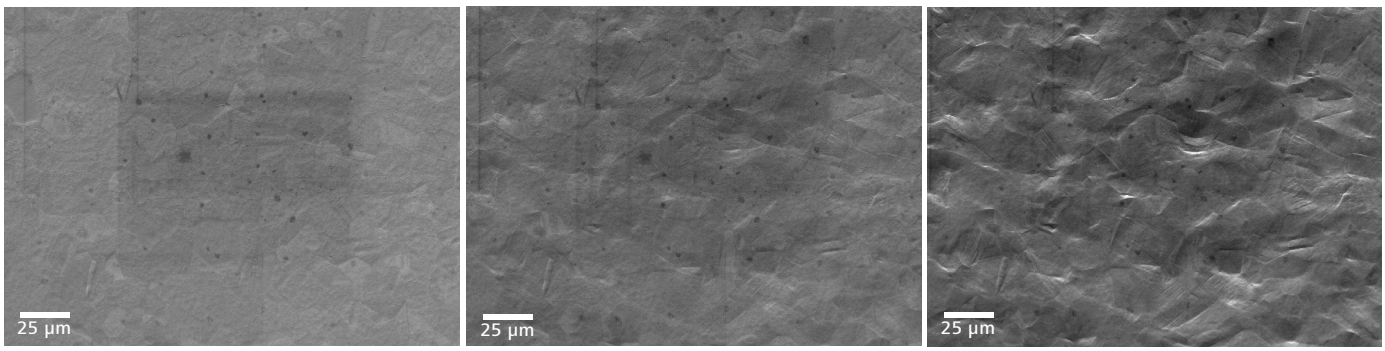


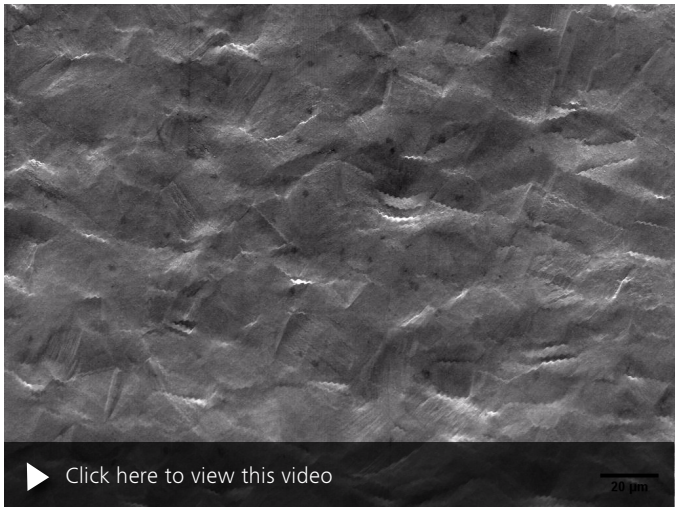
Figure 4: *SEM images of the measurement at 400 °C at 0 % (left), 5 % (center) and 10 % (right) plastic strain.*

Conclusions

From the above investigations, a clear identification of local plastic events in single grains can be observed. A series of dislocations travelling through high Schmid factor grains leads to an observable misorientation of the grain structure. These high Schmid factor grains carry the majority of the plastic deformation throughout further deformation up to plastic strains of 15 %. Observing the sample surface in the SEM while also taking grain orientation information via EBSD makes it possible to establish a correlation between these data. For the given sample material, no change in deformation behavior is observed at the tested temperatures, but the *in situ* testing method is presented as a powerful tool for investigating deformation behavior in materials science. Furthermore, the presented *in situ* method offers more data with customizable resolution along the stress-strain curve compared to conventional pre-and post-test characterization while also reducing the number of experiments necessary for achieving comparable results and saving precious sample material.

Appendix

In Videos A1 to A3 the tensile experiment at 400 °C is shown in different signal options, being SE contrast (A1), Euler Angles (A2) and IPF X coloring (A3).



***Video A1**:* In situ *tensile experiment on a X5CrNi18-10 steel up to a total plastic strain of 12 % at a temperature of 400 °C in SE contrast.*

Video A2: In situ *tensile experiment on a X5CrNi18-10 steel up to a total plastic strain of 12 % at a temperature of 400 °C in Euler contrast.*

Video A3: In situ *tensile experiment on a X5CrNi18-10 steel up to a total plastic strain of 12 % at a temperature of 400 °C in IPF X coloring.*

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