Deformation behavior study in a model dual phase system of copper–martensitic steel using in-situ synchrotron X-ray diffraction

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abstract. In this study, the deformation behavior of two phases in a model dual phase system of copper and a martensitic stainless steel was investigated using in situ synchrotron X-ray diffraction. Due to the different crystallographic structures of copper and martensite, their diffraction patterns are well separated such that the strain distribution and the load partitioning between the phases could be investigated under loading using standard methods. The copper matrix started to yield at low stress levels after a short stage of elastic deformation and the different lattice strain of the copper and martensite indicated that load partitioning took place after yielding of the copper.

1. Introduction
Dual-phase (DP) steels, typically composed of hard martensite particles dispersed in a soft and ductile ferrite matrix, show remarkable mechanical properties such as a combination of high strength and excellent formability. These properties make them attractive as engineering materials in the automobile industry and other structural applications. The mechanical behavior of DP steels arises from the partitioning of stress and strain between the two phases during deformation and depends on microstructural parameters such as grain size, distribution and volume fraction of the constituent phases. In order to understand the deformation characteristic in dual phase steels, an accurate evaluation of the micromechanical behavior of the constituent phases in such materials is required [1-4].

Synchrotron X-ray radiation can be applied to study the structural properties and deformation behavior of a wide range of engineering materials. As a powerful microstructural characterization tool, it can provide in situ analysis of the stress/strain partitioning between constituent phases and changes in lattice parameters during loading. Investigating the properties of the constituent phases is straightforward using such diffraction techniques if the two phases exhibit different crystallographic structures. However, as the diffraction patterns of the martensite and the ferrite/bainite overlap, owing to the similar crystal structures and lattice parameters of the two phases, analysis of the behavior of individual reflections is complicated and requires assumptions about the diffraction peak shapes and volume fractions of each phase [5-19].

In this article, we study the micromechanical behavior of a model dual phase steel prepared by sintering of mixed copper and AISI420 stainless steel powders and demonstrate that this material
provides a good model system for investigating the stress and strain partitioning between constituent phases.

2. Experimental
Spherical powders of copper (with a purity of 99.9%) and AISI420 stainless steel with an average size of 3.5 and 10μm, respectively, were combined in weight ratio of 70:30 using a low-energy horizontal ball milling system at room temperature. The powder mixtures were milled for 24hrs. using stainless steel balls and a polymer vial at a speed of 120 rpm with a ball-to-powder weight ratio of 20:1.

Consolidation of the mixed powders was carried out using the spark plasma sintering (SPS) process. Sintering was performed under primary dynamic vacuum with an initial pressure of 7 MPa to maintain instrument stability. A sintering temperature of 1000°C was used in this study using, heated to 950°C at a rate of 100°C/min, followed by a ramp of 50°C/min for the final 50°C heating step. After reaching the final sintering temperature the pressure was increased to a load of 50MPa and held for 5 min. The samples were then unloaded and cooled under vacuum to room temperature. After sintering, the compacted samples were heat treated in order to control the martensitic transformation of AISI420 stainless steel phase. For this, the samples were heated to 1000°C at a heating rate of 10°C/min and held for 15min before quenching in oil. The samples were then tempered in an air-furnace at 500°C for 30 min.

In order to obtain diffraction measurements with suitable time resolution and without interruption of the deformation process, the synchrotron X-ray experiment was performed at a high-flux undulator beam line (BL46XU) at the SPring-8 synchrotron radiation facility. The X-ray energy was 30keV, corresponding to a wavelength of λ = 0.413 Å, and the incident beam was 0.5 and 0.2mm in the horizontal and vertical directions. The time resolution of this experiment was 1.0s. To allow simultaneous measurements over a wide range of diffraction angles with high angular resolution, an array of six MYTHEN detectors (a one-dimensional microstrip detector made by DECTRIS) was employed [12]. The tensile tests were performed with an initial strain rate of 8.3 × 10⁻⁴ s⁻¹ at room temperature. Dog-bone shape specimens with a gauge length of 8 mm, width of 2 mm and thickness of 1 mm was used.

In order to obtain the parameters of interest such as integrated intensity, peak position and full width at half maximum (FWHM) values, the reflections of each recorded diffraction pattern were fitted using the Pseudo-Voigt function. The lattice strain distribution and evolution of different planes were investigated by analyzing the change of diffraction peak positions along 2D diffraction patterns of different phases. The lattice strain of the {hkl} planes, ε, was obtained using ε = (d - d₀)/d₀ where d and d₀ are the interplanar spacing of the {hkl} planes before and under loading, respectively, determined using Bragg’s law, λ = 2d sinθ where λ is the wavelength of the incident X-rays and θ is the diffraction peak angle. The dislocation density evolution of two phases during tensile loading was evaluated using the Williamson-Hall method:

\[
\frac{\beta \cos \theta}{\lambda} = \frac{0.9}{D} + \frac{2 \varepsilon \sin \theta}{\lambda}
\]

where β and D are FWHM and the crystallite size, respectively. The values of D and ε were obtained from the slope and intercept of a plot of β cosθ/λ against sinθ/λ for each diffraction peak. The dislocation density, ρ, was then obtained according to \( \rho = (k\varepsilon/\mathbf{b}) \) where \( \mathbf{b} \) is the Burgers vector, \( k=16.1 \) for face centered cubic (FCC) materials, and \( k=14.4 \) for body-centered-cubic (BCC) metals [20-21].

3. Results and discussion
Structural characterization was performed with the aim to determine the copper and the martensite distribution homogeneity and morphology across the specimens using a scanning electron microscope (SEM). An example SEM micrograph is shown in figure 1, where the lighter continuous phase is the copper and the darker discrete regions are the AISI420 stainless steel. Good interfacial bonding between the two phases is seen, though with some porosity, also present in the copper phase.
Figure 1. SEM image of the specimen.

Figure 2. The engineering stress-strain curve of the sample.

Figure 2 shows the engineering stress-strain curve obtained during the tensile experiment. Each point represents a set of synchrotron measurements data were simultaneously acquired for the \{111\}, \{200\}, \{220\}, \{311\} and \{222\} peaks for copper, and the \{110\}, \{200\} and \{211\} peaks for the martensite phase. The specimen was strained up to 1% strain to ensure loading into a region of macroscopic plasticity.

Example one-dimensional XRD diffraction patterns of the as-received and under a load of 335 MPa deformed samples, with the measured direction parallel to the loading direction are shown in figure 3. Before loading, the specimen exhibits phases with two types of crystal structure: a BCC martensite phase and an FCC copper phase. With increasing deformation, the intensity of copper peaks decreases and weak new peaks appear, assumed here to be the \(\varepsilon\) phase.

Figure 3. One-dimensional (1D) diffraction patterns of the model dual phase system at different applied stresses: (a) 0 MPa, and (b) 335 MPa.

Figure 4. Diffraction peaks of copper and martensite at different applied stresses: (a) 0 MPa, and (b) 335 MPa.

Figure 4 shows the effect of strain on the \{111\} and \{110\} diffraction peak for copper and martensite, respectively. The shape of the diffraction peaks under loading is unchanged, but both peaks shift towards a smaller diffraction angle. This downward shift of the peak angle represents an increase in the plane spacing in the tensile direction as a result of elastic deformation. The martensite peak is more shifted, indicating a larger tensile stress in this phase.
Figure 5. Changes in (a) copper and (b) martensite \{hkl\} lattice strains as a function of the nominal stress.

Figure 5(a,b) shows the measured lattice strains along the loading direction for different \{hkl\} planes of both the copper and martensite phases as a function of nominal stress. It can be seen that at the initial stage of loading, the lattice strains of the constituent phases increase linearly with applied stress, indicating that both phases deform elastically in this stage and different slopes are found for different reflections, indicating the different crystal orientations and anisotropy of the Young’s modulus. When the stress reaches ~25 MPa, the lattice strain in the copper phase shows a non-linear dependence on the applied stress, while the hard martensite phase continues to deform elastically, indicating a change in the stress partitioning between the two phases. It can be seen that the lattice strain of the martensite planes continuously increases during almost the entire applied deformation and the martensite bears a much higher stress than copper, which always has a lower overall lattice strain value.

The behavior of lattice strain is dependent on crystal orientation, and yielding does not take place simultaneously in all the \{hkl\} lattice planes. In the copper phase, the \{311\}, \{220\} and \{111\} planes show a similar lattice strain at each stress, though the \{311\} plane shows evidence of yielding at a lower stress than the other two planes. The lattice strain of the \{200\} planes is highest, while the \{222\} planes show the lowest lattice strain, and also show evidence of earlier plastic yielding in the copper phase. For the martensite phase, at any given stress more load is carried by the \{200\} planes than by the \{110\} and \{211\} planes.

Figure 6. Changes in (a) copper and (b) martensite \{hkl\} lattice strains as a function of the nominal strain.

Figure 6 shows the lattice strain data plotted as function of applied nominal strain along the loading direction. For the copper phase (figure 6a) it is clearly seen that plastic yielding takes place at around 0.2% strain. In agreement with the data in figure 5, the \{222\} family shows smaller lattice strains and evidence of plastic yielding at a lower strain. Figure 6b displays the evolution of lattice plane strains of \{200\}, \{110\} and \{211\} in the martensite with nominal strain along the loading direction. The \{200\} family displays the highest lattice strain while the two other families show a lower lattice strain, again reflecting the elastic anisotropy factor of the cubic crystallographic structure. All three sets of lattice
planes show a deviation from linearity (figure 6b) at around a strain of 0.4%, indicating the onset of plastic flow in the martensite phase. The lattice strains measured of the martensite reflections are all larger than those for the copper phase. The data show that for both phases the lattice strains exhibit two different stages of deformation. The first linear region corresponds to the elastic loading in both the copper and martensite. With increasing applied strain, the copper matrix starts to yield, while the martensite continues to undergo elastic deformation. The difference of lattice strain in copper and martensite indicates that load partitioning takes place after yielding of the copper. With further loading the lattice strain of the copper phase shows a gradual increase while the lattice strain in the martensite continues to increase linearly until around 0.4% applied strain, at which point plastic yielding in the martensite takes place. A limited work hardening can be seen in martensite phase, whereas copper phase shows work hardening throughout the entire loading.

![Dislocation density evolution in (a) copper and (b) martensite as a function of the nominal strain.](image)

The pattern of load distribution between the two phases are also seen in the calculated changes in dislocation density with increasing nominal strain during tensile deformation, illustrated in figure 7. Before loading, the martensite phase already contains a moderate dislocation density of about $2.6 \times 10^{16}$ m$^{-2}$ compared to a value of $9.5 \times 10^{15}$ m$^{-2}$ in the copper phase. As the loading begins, extending from strains of 0% to about 0.11%, which is the region of elastic deformation, the dislocation density shows no change in copper phase. After it has entered the fully plastic flow stage, the dislocation density increases continuously with increasing strain up to $1.5 \times 10^{15}$ m$^{-2}$ before unloading, showing work hardening in the copper phase. In contrast, the dislocation density of the martensite phase shows a wide scatter and increases only slightly throughout the investigated load range, with a maximum dislocation density in the martensite during loading of $3.5 \times 10^{16}$ m$^{-2}$.

4. Summary

The micromechanical behavior of a modal dual phase steel and the relationship between microstructure and mechanical properties of its two phases have been investigated by synchrotron X-ray diffraction during tensile deformation at room temperature. The use of a model dual phase system represents a novel approach for fundamental studies of the deformation behavior of dual phase steels as a full separation of the different peaks from the hard martensite and soft copper in X-ray diffraction measurements provides data on the load transfer between the two phases without the need for peak deconvolution methods and related assumptions about the diffraction peak shapes. Moreover, the use of powder mixing and sintering allows independent control of both the size and volume fraction (and potentially the hardness) of each phase, thereby facilitating exploration of optimized microstructural combinations.

At low applied stresses, elastic loading of both phases was found to occur. At higher stresses, macroscopic yielding of the copper matrix took place, the load partitioning among phases was observed and the hard martensite bore significantly larger load than the soft copper matrix. In the copper matrix, the (222) family showed the lowest lattice plane strain and earlier yielding. Both phases had some dislocations before loading, but it is much higher in martensite and increased slightly
throughout the loading process. The dislocation density in the copper phase started to rise after entering the plastic stage and increased sharply before unloading showing work hardening in this phase while the martensite phase has just entered the plastic deformation region.

5. References

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