Short Communication

Effect of the gold remodeling preparation method on the microstructure and mechanical behavior of steel

Christian Oen Paulsen1,2 · Tore Børvik2,3 · Ida Westermann2,4

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Abstract

The application of gold speckles on a polished surface is a successful technique for improving digital image correlation (DIC) contrast in scanning electron microscope (SEM) images. In the process of creating the gold speckles, the material is subjected to elevated temperatures for prolonged times. As a consequence, not all materials are suitable for the gold speckled method to improve the contrast for DIC measurements during an in-situ SEM tensile test. In this letter, the effect of gold remodeling on two different steels is investigated. These steels are a dual-phase (DP) steel and a ferrite–pearlite steel (NVE36). The results demonstrate that the temperature these steels are subjected to during gold remodeling will influence the tensile behavior of the DP steel while the NVE36 steel is unaffected by the heat treatment. As a result, we can conclude that the gold remodeling method for creating contrast in SEM images may affect the microstructure. However, the effect of these changes depends on the material at hand and will vary from material to material.

Keywords
Digital image correlation (DIC) · Gold remodeling · Heat treatment · Scanning electron microscope (SEM) · In-situ tensile testing

1 Introduction

One main challenge in scanning electron microscope (SEM) imaging when performing in-situ tensile tests is to achieve sufficient contrast to obtain the material’s strain field during deformation based on digital image correlation (DIC). A successful technique has been to apply a gold speckled pattern on a polished surface. For instance, Orozco-Caballero et al. [1] achieved a spatial resolution of 44 nm in their strain fields using this technique. The method for remodeling a thin continuous layer of gold into a speckled pattern requires an elevated temperature during the remodeling process. Here, an investigation is conducted in order to determine the effect of this heat treatment on the properties and microstructure of the steels studied. These materials are DP500 (a ferrite–martensite steel) and NVE36 (a ferrite–pearlite steel). Each of them was characterized before and after the heat treatment process by using scanning electron (SEM) micrographs, electron backscatter diffraction (EBSD), hardness tests and tensile tests. The main objective of this study is to investigate the effect of temperature on the mechanical response during the gold remodeling. This paper illustrate the need for this type of study to be carried out when heat treating metallic materials to achieve a gold speckled pattern for the DIC analysis on SEM images.
2 Material and methods

The objective of this study is to investigate the effect of temperature during the gold remodeling on two different steels. In order to remodel the continuous gold layer into gold speckles, a method in which the specimens are exposed to 180°C for 96 h in an argon/styrene atmosphere is utilized on both steels. This method is described in further detail in [1]. The resulting strain field from the steels are seen in Fig. 1. These maps have a spatial resolution of 96 nm and 140 nm for the NVE36 and DP500, respectively. This is in contrast to a spatial resolution of 2240 nm achieved by just etching the microstructure of NVE36 without adding gold [2].

Both materials investigated in this study were extracted from rolled steel plates containing two phases, each exhibiting a greatly different hardness, strength and ductility. DP steels contain ferrite and martensite. Martensite is a hard and brittle phase, while ferrite is a ductile phase. The chemical composition of the DP500 is: 0.079 wt% C, 0.7 wt% Mn and 0.3 wt% Si. NVE36 consists of ferrite and pearlite. Pearlite has a lamellar structure, where the lamellas are alternating between cementite and ferrite. Cementite is a hard and brittle phase, while the ferrite is a ductile phase. By altering the ratio between the two phases, different combinations of strength and ductility can be achieved for both DP500 and NVE36. The chemical composition of NVE36 is: 0.18 wt% C, 0.5 wt% Si, 0.9 wt% Mn, 0.2 wt% Cr, 0.4 wt% Ni, 0.35 wt% Cu, 0.035 wt% P and 0.035 wt% S.

All specimens were investigated before and after the heat treatment. Microhardness measurements, based on seven different indentations, were taken from each phase. A small load of 10 g, with a holding time of 15 s was used to fit the hardness imprints within the grains. The measurements were performed using a Leica VMHT MOT microhardness tester on polished specimens. All specimens were ground and polished to 1 μm before the final step to reveal the microstructure. For the DP steel, the final step was vibration polishing by a VibroMet2 from Buehler for 12 h using a suspension with pH 8 containing 0.02 μm SiO2 particles. NVE36 was etched with 2% Nital for 10 s. Finally, both specimens were cleaned in an ultrasonic bath for 5 min with acetone.

In addition to the hardness measurements, EBSD scans were acquired for the DP specimen to disclose the grain size and phase composition of the material. The specimen was prepared for the EBSD acquisition in the same fashion as before the hardness measurements. During indexing of EBSD data, the martensite was indexed as ferrite with poor correlation. The martensitic phase was then identified by using the image quality (IQ) map in the EBSD software. For NVE36 it was difficult to identify the pearlite in EBSD maps, but the pearlite grains were visible in secondary electron (SE) imaging mode on the etched surface. As a consequence, the grain size of ferrite and pearlite, and the phase composition of NVE36, were identified using microstructural SE images instead of EBSD scans as for the DP steel. The SEM was chosen to record and characterize the microstructure over micrographs recorded in light microscope since it was found that the contrast between phases and the contrast on grain boundaries were suboptimal.

A total of three maps from both EBSD and SE images were used for each specimen to identify the grain size. All maps had an area of 80 μm × 80 μm and included

![Fig. 1](image-url) DIC maps of the NVE36 and DP500 steels showing the major principle strain. Here, a has a spatial resolution of 96 nm, b has a spatial resolution of 140 nm. The pulling direction of the in-situ SEM tensile test on both specimens is indicated in the bottom right of b.
hundreds of grains in each. These images were acquired from random areas from the specimen surface and it is not the same area recorded before and after heat treatment. In addition, tensile test curves were acquired before and after the gold remodeling for all specimens. These tensile tests were performed using an MTS810 100kN conventional tensile test machine using DIC as a virtual extensometer (see Paulsen et al. [2] for the specimen geometry). All tensile tests were carried out using a constant strain rate of 1.11 × 10^{-3} \text{s}^{-1}.

The results from the hardness measurements and the microstructure statistics from the EBSD scans and SE images, both before and after the heat treatment, are listed in Fig. 2a–c. These figures show the results from all measurements acquired in this work and compare the values before and after the heat treatment. Only small discrepancies between the measurements are seen. All measurements taken after the heat treatment are within the standard deviation of the measurements from before the heat treatment. The only exception is for the martensite grain size. The difference between 4.3 ± 0.75 and 3.2 ± 0.15 \mu m is great in terms of percent difference, but low in absolute terms.

In Fig. 3 the engineering stress–strain curves for the different specimens, before and after the heat treatment, are shown. There is a clear difference in the curves for the DP500. Here, the specimens exhibit a sharp yield point and a yield plateau with Lüders band propagation before the work hardening starts after heat treatment. In addition, the yield strength is increased by roughly 100 MPa. However, the work hardening after passing of the Lüders band in the DP steel is similar before and after the

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![Fig. 2](image-url) **Grain size, hardness and phase composition before and after heat treatment**

![Fig. 3](image-url) **Stress–strain curves for both materials before and after heat treatment**
heat treatment. The tensile curves for NVE36 are close to identical before and after heat treatment. Here, the small differences in the curves can be ascribed to the natural variation in the material.

3 Discussion

In this work, two different steels have been heat-treated to replicate the conditions during the remodeling of a continuous layer of gold into a speckled pattern. The speckled pattern can be used to correlate a DIC analysis from backscatter electron (BSE) images acquired during in-situ SEM tensile testing. When studying the microstructure and hardness measurements before and after heat treatment, it seems as though no changes have taken place in any of the steels investigated. The before and after columns in Fig. 2a–c are close to equal and within the standard deviation for all specimens. Also, from visual inspection of micrographs, it is not possible to distinguish which microstructure has been heat-treated and which has not.

In addition to the microstructural investigations, tensile tests were performed on all steels before and after heat treatment to reveal any changes in the mechanical response. The NVE36 specimens indicated no difference between the heat-treated tensile curve and the as-received tensile curve (Fig. 3b). However, this is not entirely unexpected. The NVE36 microstructure is decided by the metastable Fe-C phase diagram. As a result, NVE36 should withstand 180 °C for an infinite amount of time without any changes to the microstructure.

However, looking at the tensile curves for DP500 in Fig. 3a, it is clear that some changes have taken place. After the heat treatment, a sharp upper yield point and a yield plateau with Lüders band propagation have been introduced. In addition, the yield strength has increased significantly. These changes are due to static strain aging, also known as bake hardening [3]. The fundamental mechanisms behind the bake hardening phenomena were studied by Waterschoot et al. [4]. After tempering for 5 h at 170 °C, clear signs of η-carbides precipitation were observed. Also, there were strong suggestions of relaxation of internal compressive stresses. This might help to explain the increased yield strength since the carbon has diffused into the ferrite and precipitated as η-carbides. However, apart from the rearrangement of carbon, the microstructures are close to identical (as observed in Fig. 2a–c). As a result, during hardening (after passing of the Lüders band) the heat-treated curves follow the same trajectory, i.e., the same work-hardening, as the as-received material.

4 Concluding remarks

The main motivation for this work was to determine if the remodeling temperatures required to remodel a continuous layer of gold into a speckle pattern influenced the microstructure and mechanical properties of the investigated materials. Each specimen was heat-treated to imitate the gold remodeling process, with the same temperature and holding time. All micromechanical parameters measured and shown in Fig. 2a–c remain within the standard deviation of the measurements and seems unaffected by the heat treatment. However, for DP500 steel the stress–strain curve from before and after the heat treatment differed at low strains. Here, the yield strength increased and a Lüders plateau was introduced. In contrast, NVE36 behaved identically as before the heat treatment during a tensile test after the heat treatment. As a consequence, NVE36 is very suitable for gold remodeling at 180 °C for 96 h. However, martensitic materials, like DP500, are less suitable since alterations in the mechanical response may occur. This is especially the case for the onset of yielding in the material. The rate of hardening after the Lüders plateau is similar before and after being heat-treated for all DP steel specimens. As a result, during the hardening process, this method is still useful since the hardening behavior seems identical before and after the heat treatment. The heat treatment only affected some type of materials and mainly at small plastic strains. In studies of ductile fracture, which takes place after considerable plastic strain, the effect of the heat treatment on the mechanical response seems to vanish.

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Compliance with ethical standards

Conflict of interest All authors declare that they have no conflict of interest.

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