

Solid State Phase Transformation Mechanism in High Carbon Steel Under Compressive Load and with Varying Cr Percent

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Abstract Low alloyed High carbon steels with duplex (DHCS)s structure of martensite and retained austenite (RA) have considerable potential for industrial application in high abrasion environments due to their hardness, strength and low cost. Using standard compression testing, XRD, nano-indentation, EBSD and TEM, we determined the mechanical stability of RA in DHCS under compressive stress and recognized the phase transformation mechanism, from the macro to the nano level. We found that at the initial stage of plastic deformation both BCT and HCP martensite formation takes place, whereas higher compression loads trigger BCT martensite formation. The combination of this phase transformation and strain hardening is able to increase the hardness significantly. We also investigated the effect of Cr on the transformation behaviour, hardness and mechanical stability of RA with varying Cr contents. Increasing Cr% increased the stability of retained austenite, consequently, increased the critical pressure for martensitic transformation.

Keywords High carbon steel · Solid-state phase stability · Retained austenite Effect of Cr

Introduction

High carbon steel with the martensite and retained austenite (RA) structure is favourable for the extreme operation condition and high abrasion environment due to its high hardness and relatively low cost. The amount and stability of retained austenite in the martensitic steel plays an important role in optimizing the mechanical properties. Changing chemical composition [1], strain partitioning [2], formation of mechanical twinning [3], grain size refinement [4] and martensitic

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transformation [1, 5] due to the solid state compression deformation are linked to the stability of retained austenite and mechanical property of the steel. In this steel, metastable RA transforms to the stable martensite structure when it can pass the required barrier energy. This energy can be achieved through, stress, strain and/or temperature [2, 6]. The strength and ductility of this steel is affected by the phase transformation and strain hardening effect through deformation [7, 8].

Stability of RA is studied by a lot of researcher; however there are few studies in the literature focusing on the stability of retained austenite in the low alloyed high carbon steel, DHCS. The present work is aimed at investigating the mechanical stability of retained austenite grain from macro to nano scale by the standardize compression test on bulk material and nanoindentation technique in individual retained austenite (RA) grains, combined with electron backscattered diffraction (EBSD), X-ray diffraction (XRD), focused ion beam (FIB) milling and transmission electron microscopy (TEM).

Results and Discussion

We investigated 1.0% C DHCS with a mixture of plate and lath martensite and containing ~45–50% retained austenite. Figure 1a shows the EBSD micrograph of the undeformed sample where BCT martensite is shown in red, HCP martensite in yellow and the retained austenite in blue in the phase diagram. The black lines on the maps represent the boundaries, across which misorientation is more than 15°. These EBSD scan were conducted with a pattern binding of 2×2 , with an integration number of frames of 10 for 25 kV and that the step size chosen was 50 nm. The kernel average misorientation (KAM) of the undeformed steel sample is also presented here which shows higher misorientation in the close vicinity of martensite boundaries.

We designed a standardize compression test in the small sizes (4mm \times 4mm \times 4mm) samples at room temperature with an Instron 8510 instrument operating at 0.10 mm/min cross-head speed. Figure 1b, c shows the EBSD results for the samples at 2000 and 3500 MPa compression. The results revealed both the transformation of RA to martensite and the generation of smaller martensitic grains. The corresponding XRD patterns revealed that, after deformation, there are variations in the peak intensities and a deformation-induced phase transformation occurred as evidenced by a more prominent BCT α' -martensite peak and a reduced γ -austenite peak. It is worth noting that at moderate compression deformation, both BCT α' -martensite and HCP ϵ -martensite formation triggered. However, at larger strain due to higher compressive load only BCT α' -martensite formation dominates. More misorientation was observed while the sample was induced to more load. As the compression load increases the misorientation angle becomes high enough to form new grain boundaries which further reduces the misorientation in the microstructure (Fig. 1b2, c2). The sample hardness increases with increasing martensitic volume fractions because the martensitic structures act as a barrier to

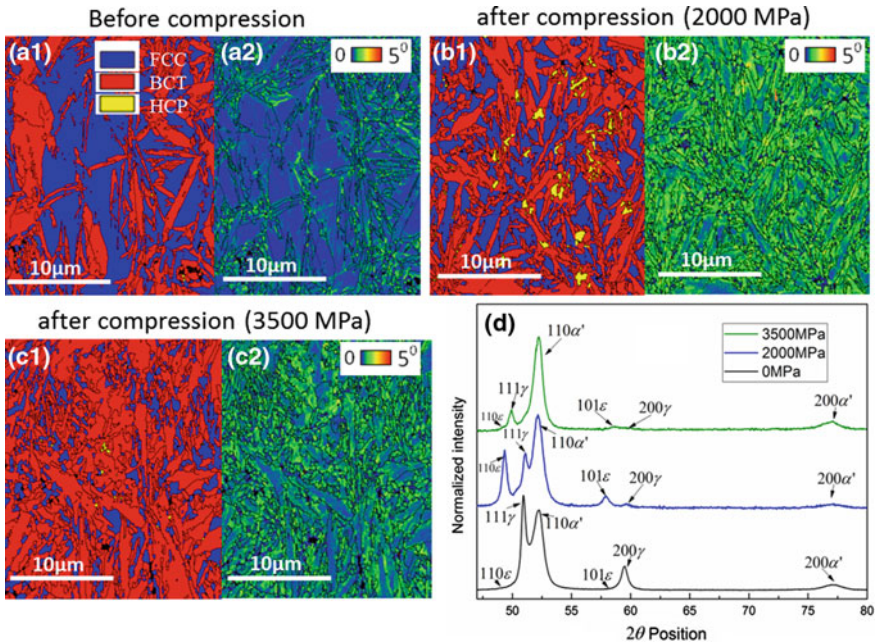


Fig. 1 EBSD patterns of studied materials; Phase map and KAM (a) without compression and compressed at b 1000 MPa and c 3500 MPa. d XRD spectrum of sample with and without compression deformation

dislocation movements. Refined grains also can strengthen the microstructure. Before compression deformation the overall hardness measured in the sample was ~ 7.51 GPa which was increased after compression deformation and calculated to be ~ 9.76 GPa in a micro Vickers hardness tests performed at 0.2HV load.

A nano indentation investigation on the individual austenite grain was performed to identify the phase transformation phenomena at the nano level. Nano-indentation tests were carried out in load control mode on a TI 900 Hysitron Tribolab system with a Berkovich three-sided pyramidal diamond tip indenter with a tip radius of 100 nm. It is possible to detect the position of each indent by EBSD micrograph, Fig. 2a shows that after the indentation the austenite grain was transformed to martensite within the close vicinity of the indentation mark and the transformation process was observed by several pop-in as marked with arrows in the P-h curve (Fig. 2b). After using focused ion beam (FIB, XT Nova Nanolab 200, at 30 kV) to prepare the sample a further TEM micrograph and SAD pattern by a Philips CM 200 with a field emission gun reveals clear martensitic transformation due to the indentation load. It is also possible to identify the critical force (P_c) for the martensitic transformation at the nano level due to the indentation. The first pop-in in the elastic plastic regime of the P-h curve shows the critical load for the

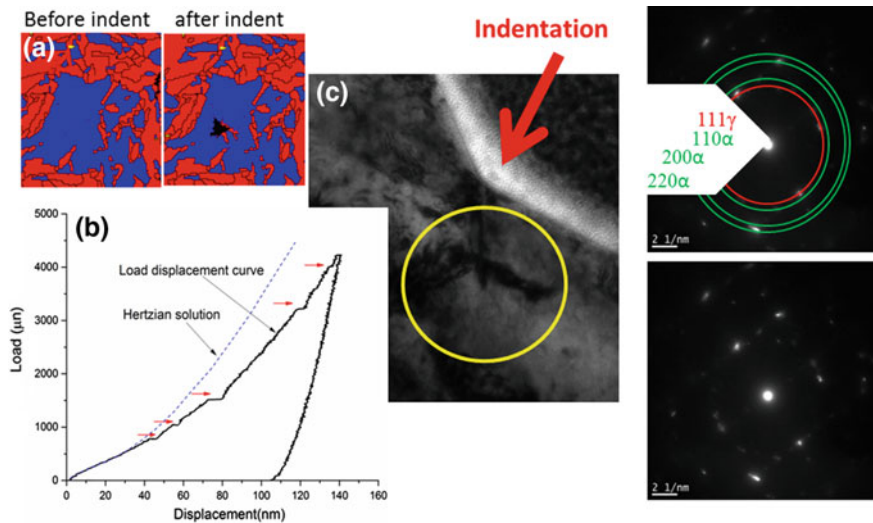


Fig. 2 **a** The EBSD phase map of austenite grain before and after nano-indentation. **b** Nano-indentation load displacement curve of corresponding indent. The red arrows are indicating pop-ins during nano-indentation and blue dashed line represents the calculated Hertzian elastic contact solution. **c** Bright field TEM image and SAD pattern showing existence of α' martensite and retained austenite at spot

martensitic transformation which is an indicative of the stability of retained austenite at the nano level [5, 9].

Some of the elements within steel have the effect on the stability of the retained austenite, i.e. C, Mn, Cr [5, 10] etc. This research investigated steels with varying chromium contents in chemical composition. To keep the steel cost effective for industrial applications we varied the Cr percentage within a smaller range to design and characterize low cost low alloyed high carbon steel. Chromium addition caused an increase in the eutectoid temperature and a drop in the eutectoid carbon content. Therefore, Manganese content was adjusted to offset these effects. By adding Cr it is possible to prevent the corrosion and oxidation and enhance the higher temperature strength [11]. For this purpose we designed three steel samples with varied Cr% within the range of 0.10–2.5%. The samples selected had similar retained austenite in order to establish the effects of chromium on the solid-state transformation of retained austenite. Nano indentation load displacement curve of individual austenite grains from 3 samples are presented in Fig. 3. The results revealed that the increased Cr% contributed to the higher mechanical stability of the austenite grains; i.e. the sample having lower Cr% (0.1–0.18% Cr) has lower critical load which was $\sim 900 \mu\text{N}$ and for the higher Cr sample (0.6–0.8% Cr), it was $\sim 6000 \mu\text{N}$. At the same nano-indentation load, 8000 μN , the hardness measured 8.3, 7.5 and 6.1 GPa for low (0.1–0.18%), medium (0.6–0.8%) and high (1.8–2.5%) Cr samples within this short range. For the increased Cr% the austenite grain resists more for the transformation process. In the lower Cr sample, the stability of the retained

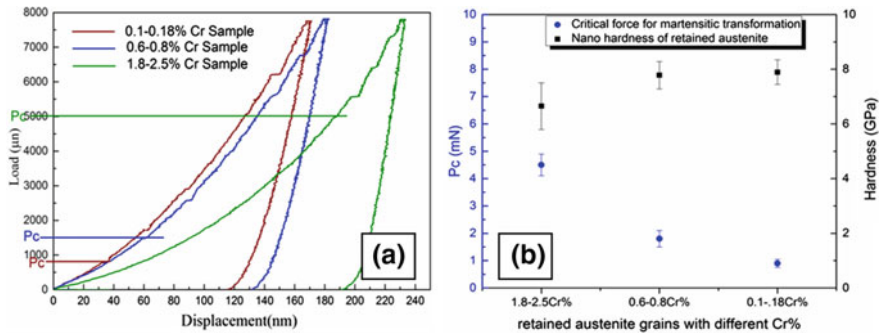


Fig. 3 **a** Load displacement curves by nano indentation in austenite phases for three samples. P_c indicates the critical load for martensitic transformation. **b** The average critical loads for martensitic transformation and corresponding hardness of austenite phases in the samples. This statistics are the summarization of atleast 50 indents on austenite phase for each samples

austenite is less and it transforms more to the martensite. As martensite is harder than the austenite phase, more martensitic transformation leads to elevated hardness. This is the reason behind the hardness variation in the retained austenite with varied Cr%. In this study, the average hardness of retained austenite was calculated 6.95 GPa in sample with 1.8–2.5% Cr; 7.78 GPa in sample with 0.6–0.8% Cr and 7.89 GPa in sample with 0.1–0.18% Cr respectively by the nano indentation investigation. Nevertheless, the actual hardness of austenite should be lower. Martensitic transformation increased the nano-hardness of retained austenite.

In the current study we have described comprehensively the effect of external compressive stress on martensitic transformations and grain size refinement, effect of Cr% on retained austenite stability within a small range. Such understanding is critical for controlling the microstructure, hardness and mechanical stability of DHCS to design it as a low cost low alloyed steel for industrial applications.

Conclusion

In the present work, the mechanical stabilities of metastable retained austenite in high-carbon steel were investigated by using a combination of standardize compression test, nano-indentation, EBSD, FIB and TEM. The results revealed that, it is possible to increase the hardness of DHCS through the combination of phase transformation, grain refinement and strain hardening by compression deformation. The effect of varied Cr% was also described which demonstrated that by increasing the Cr%, the stability of retained austenite was increased, consequently, the critical pressure for martensitic transformation was also increased. This study makes a significant and important contribution to our understanding on DHCS, thereby potentially opens new scopes of applications and replace more expensive high alloy

grade steels. This could increase the usage of cost-effective high carbon steels in industry, with significant flow on benefits for businesses and economies.

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