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Influence of nitrogen enrichment on the austenite grain growth and decomposition upon cooling in low-alloy steels by in-situ characterization

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Abstract

Thermochemical surface treatments, such as carburizing are widely used methods for case hardening in order to improve wear resistance, impact strength, and fatigue properties in gear applications. Alternatives to carburizing are nitriding or carbonitriding by diffusing nitrogen into the surface of metals for further improvement of the properties, whose physical origins are yet to be established, especially regarding microstructure formation. Previous studies^[1] showed that nitrogen enrichment in the austenite has large consequences on its decomposition. The kinetics are largely accelerated and the resulting microstructures are much finer, and associated with stronger properties (hardness)^[2]. These effects have been ascribed so far to the formation of nitrides (mostly CrN, MnSiN₂) in the austenite. These would provide new nucleation sites for the ferrite. In addition, nitrides could also contribute to austenite grain refinement upon re-austenitization. This could be another origin of the acceleration of kinetics and the microstructure refinement. In this work, new EBSD observations demonstrating the refinement of the austenite grains are presented. A suitable experimental approach is then introduced to study the refinement of the austenite, thanks to in-situ microscopy techniques: high-temperature in-situ EBSD and laser scanning confocal microscopy (LSCM). Previous results from in situ HEXRD showing the acceleration of the austenite decomposition are also recalled.

Experimental details

Samples with a thickness of 0.5 or 1 mm of a steel containing 0.25C - 1.21Mn - 1.31Cr - 0.24Si - 0.18Ni (wt.%) were homogeneously enriched in the austenitic phase field at 900°C with carbon and / or nitrogen by cracking respectively methane or ammonia using a laboratory thermobalance^[3]. The enrichment treatment was followed by oil quenching to room temperature. The concentrations of carbon and nitrogen in solid solution as measured by an electron probe micro-analyzer (EPMA) are reported in Table 1.

Table 1. Range of composition obtained upon EPMA analysis of the enriched steels

Steel	Reference	wt.% C	wt.% N
Base	I	0.25	0
Carburized	C	0.4 – 0.7	0
Nitrided*	N	0.1 – 0.3	0.2 – 0.5
Carbonitrided	C+N	0.4 – 0.7	0.2 – 0.5

*Steel has undergone decarburization during nitriding.

The decomposition kinetics of the austenite were studied under isothermal conditions (400°C) after re-austenitization at 900°C for 10 min, by dilatometry and by HEXRD synchrotron in-situ (at the ESRF)^[1,3]. Other microstructures obtained upon re-austenitization and fast continuous cooling (~100°C/s) were examined by SEM equipped with EBSD. In-situ optical and EBSD studies were conducted during continuous reheating experiments on the enriched samples using a confocal laser scanning microscope and high temperature EBSD set-up, respectively. In-situ HEXRD measurements are also being conducted presently during continuous cooling using the synchrotron radiation at DESY.

Effect of nitrogen enrichment on kinetics of austenite decomposition : in-situ HEXRD

Figure 1 shows the kinetics of bainitic ferrite formation during isothermal treatment at 400°C after having re-austenitized the enriched samples^[1]. Results for the base steel (I) are also plotted for comparison. The I steel shows the fastest kinetics of ferrite formation whereas after carbon enrichment (C steel), there is a drastic reduction of the kinetics, as expected. The effect of nitrogen is highlighted by comparing C and C+N steels: despite similar higher content of austenite stabilizing interstitials, the kinetics is much faster in C+N steel. Exhaustive microstructure observations also revealed a strong refinement of the microstructure^[1-4]. In these articles, these effects were ascribed solely to the enhancement of the bainitic ferrite nucleation by the nitrides which precipitated during the enrichment in the austenite field.

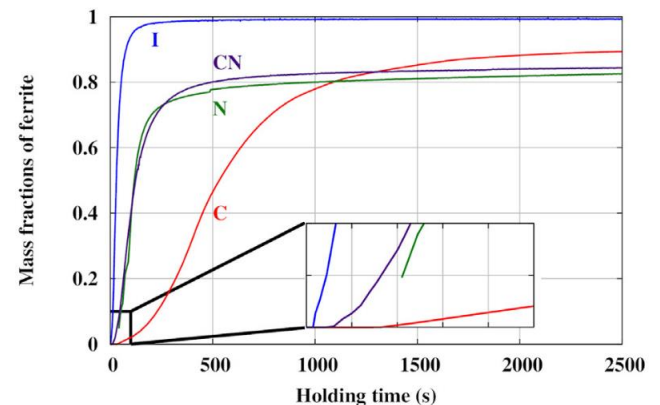


Figure 1: Evolution of the mass fraction of bainitic ferrite (with a zoom for short times) during re-austenitization and isothermal treatment at 400°C, for different enriched samples from in-situ HEXRD^[1]

Austenite grain refinement: EBSD results

Austenite grain refinement upon reheating after the enrichment treatment is another possible origin of these kinetics and microstructural effects. Evidences of austenite grain refinement are presented here. The austenite being a high-temperature phase, it is difficult to directly observe its grain structure at the room temperature. EBSD-based crystallographic reconstruction techniques have been employed to obtain the austenite grain structure^[5–7]. As-enriched as well as re-austenitized and quenched microstructures have been analyzed by EBSD to acquire orientation maps of the as-quenched bcc (martensite or a mix of martensite + bainitic ferrite) phases. The prior austenite grain structure was then reconstructed, using continuous optimization and refinement of the Kurdjumov-Sachs α/γ orientation relationships. Figure 2 a, b show the reconstructed austenite grain structure in the as-enriched C and C+N sample before re-austenitizing. The austenite grain structures after re-austenitizing to 900°C for 10 minutes and quench are shown in Figure 2 c, d. The prior austenite grain size is found to be $\sim 16 \pm 3 \mu\text{m}$ for all the as-enriched samples prior to reheating. There is no effect of the steel interstitial content. After re-austenitizing and quench, the carburized sample retains similar grain size. Conversely, there is a strong austenite grain refinement for the carbonitrided sample. This was also the case for a nitrided sample (not shown here). In addition, several micro-sized CrN precipitates are identified at the prior austenite grain boundaries of the nitrogen enriched samples along with fine intragranular precipitates.

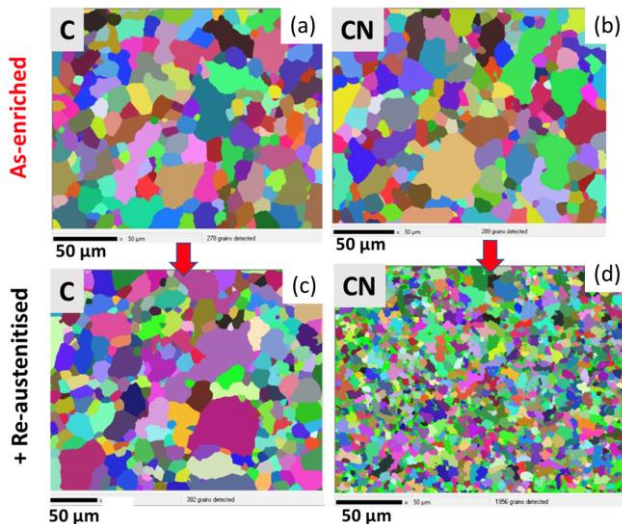


Figure 2: Evolution of the parent austenite grains upon reconstruction from EBSD data for the C, N and C+N samples in the as enriched state and upon re-austenitizing (the γ grains – including twins – are random colored)

These results may suggest that the CrNs and other nitrides may pin the austenite grain boundaries restricting the grain growth of austenite and/or enhance austenite nucleation. Further confirmation could be obtained from in situ microscopy techniques using the hot stage inside SEM coupled with CMOS based EBSD camera. High indexing rate and acquisition speed were obtained at 900°C during preliminary experiments using that set-up.

In-situ Laser Scanning Confocal Microscopy

A continuous heating at 3°C/s up to 1300°C was applied to examine the nucleation-growth of the austenite inside a C+N steel sample. The high maximum temperature allows to observe the entire grain growth regime. The results indicate that the formation of austenite is completed at about $\sim 800^\circ\text{C}$. The grain growth kinetics is slow up to 1100°C with an average grain size of 11 μm weighted by the area, after which rapid acceleration of γ grain growth occurs resulting in a grain size of $\sim 17 \mu\text{m}$ at 1300°C. Possible correlation with CrN dissolution is currently being analyzed.

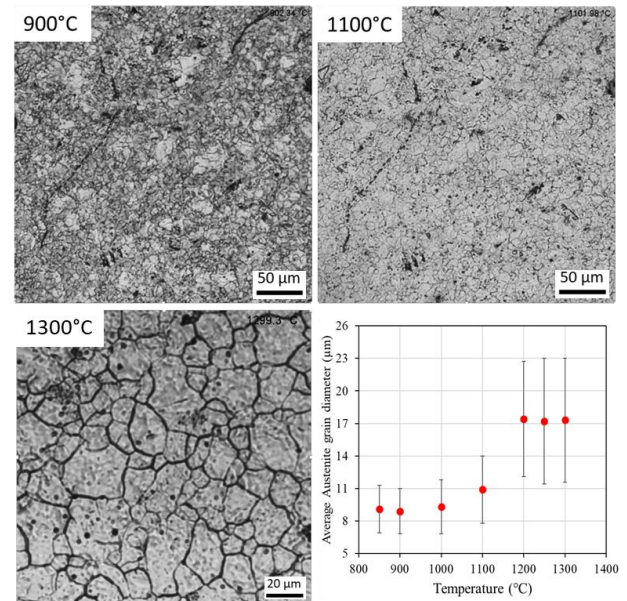


Figure 3: Austenite grain growth observed in a carbo-nitrided sample (0.45C, 0.35N, in wt.%) during continuous reheating to 1300°C at 3°C/s measured by in-situ LSCM

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