## Characterization of Products of Competitive Reactions during Quenching and Partitioning Applied to a Ductile Cast Iron

Competitive reactions have been reported to occur during critical steps of the Quenching and Partitioning process, leading to very complex microstructures. In this work we have characterized the products of competitive reactions – namely transition carbides and bainitic ferrite – by SEM, EBSD, and 3D-APT. A methodology for determining the accurate orientation relationship between fcc and bcc phases has been successfully implemented.

The Quenching and Partitioning (Q&P) process applied to ductile cast irons has been proposed as an alternative heat treatment to austenite-containing microstructures obtain similar to those found in the austempered ductile cast iron (ADI) [1][2]. The Q&P process involves a partial quenching of the material to produce a controlled mixture of martensite  $(\alpha')$  and untransformed austenite ( $\gamma$ ), followed by a step, when the material is "partitioning" isothermally held at a higher temperature to promote the carbon diffusion from the carbon-supersaturated martensite to austenite. The carbon-enriched austenite is stabilized by the high carbon content and does not transform into martensite during the final cooling. In the final microstructure, martensite confers high strength, while stabilized austenite favors good ductility due to occurrence of TRIP effect [1][3]. Competitive reactions, such as carbides precipitation in martensite and decomposition of austenite to bainite, are reported to occur during the partitioning step, leading to very complex microstructures. Addition of large fractions alloying elements (e.g., Si and Mn) has been proposed to avoid such reactions, but processing difficulties (e.g., microsegregation) have been reported to arise from this practice [4].

A low Mn ductile cast iron alloy (Fe - 3.47C -2.47Si - 0.20Mn - 0.38Cu, wt.%) was submitted to Q&P heat treatments at various conditions was previously characterized by SEM, EPMA, dilatometry, and in-situ synchrotron X-ray diffraction. Austenitization was carried out at 880 °C in the v + graphite phases field and initial austenite carbon composition is approximately 0.8 wt.%. Formation of bainitic ferrite ( $\alpha_{b}$ ) was observed by in-situ XRD and has been pointed out as the main mechanism of austenite carbon-enrichment. Evidence of transition carbides precipitation was obtained by analysis of EPMA and in-situ XRD results. The aim of this collaborative work was to identify the critical heat treatment steps were carbides precipitation occurs, to understand the distribution of retained austenite and fresh martensite. To achieve this goal, experimental techniques performed at IMR encompassed SEM, EBSD, and 3D-APT.

SEM characterization shows presence of fine dispersion of carbides inside martensite plates after very short partitioning times (< 30 s), as



Fig. 1: SEM and EBSD phase maps of Q&P treated samples. Phase colored as red were indexed as bcc, while phase colored as green were indexed as fcc. a) and b): quenching at 170 °C, partitioning at 375 °C for 30 s. c) and d): quenching at 170 °C, partitioning at 375 °C for 15 min.

shown in Fig. 1a. EBSD characterization reveals localized distribution of retained austenite after the Q&P heat treatment. For short partitioning times (Fig. 1b), large regions that did not transform into martensite or bainitic ferrite up to the end of the partitioning step transform into fresh martensite during the final cooling. mainly Retained austenite is observed surrounding martensite and bainitic ferrite plates/laths. Longer partitioning times (Fig. 1c-d) lead to a higher fraction of bainitic ferrite, and a more homogeneous distribution of austenite, while formation of fresh martensite is ceased.

The microstructure of a sample partially quenched but not subjected to the partitioning step presents etching features that resemble carbides. 3D-APT characterization of the same sample (Fig. 2a-b) shows presence of carbon clusters achieving maximum carbon content of 15 at.%, much lower than the stoichiometric composition of cementite (25 at.%) or transition carbides (e.g.,  $\varepsilon$  carbide, 25-33 at.%). From these results it is proposed that carbon clusters formed during the quenching step act as nucleation sites for precipitation of transition carbides during the reheating and partitioning steps of the Q&P heat treatment.



Fig. 2: 3D-APT analysis of sample quenched at 170 °C for 1 min then cooled to room temperature. a) Isoconcentration (4 at.%) surfaces of carbon composition. b) Carbon proxigram obtained on the volume highlighted in Fig. 2a.

Accurate orientation relationship between the bcc (martensite and bainitic ferrite) and the fcc (austenite) products was also determined from the measured EBSD data [5]. In Fig. 3a the deviation of the accurate orientation relationship from Kurdjumov-Sachs orientation the relationship (KS-OR) in a Q&P treated sample is represented in color map. Differences of deviation angle are observed inside single bcc microconstituents plates/laths. Fig. 3b shows the same information obtained for a material containing only bainitic ferrite and austenite (produced by austempering). The bainite product in this microstructure is coarser and the deviation of the accurate OR from the KS-OR is notably higher than in the Q&P sample. Since in the Q&P sample bainitic ferrite is nucleated at martensite/austenite interfaces, it is suggested that the martensite/austenite OR plays an important role defining the bainitic ferrite/austenite OR.

In summary, we have conducted extensive characterization work on Q&P multiphase microstructures. Valuable information about phase distribution was obtained by SEM and EBSD analysis. A methodology for determining the accurate orientation relationship between bcc and fcc phase has been successfully implemented for the current set of microstructures.



0.0 0.5 1.0 1.5 2.0 2.5 3.0 3.5 4.0 4.5 5.0 Deviation angle of accurate OR in relation to KS-OR (deg.)

Fig. 3: Deviation angle of the measured accurate OR in relation to KS-OR. a) Sample austempered at 375 °C for 15 min ( $\alpha_b$  +  $\gamma$  microstructure). b) Q&P sample quenched at 170 °C and partitioned at 375 ° for 30 s.

## **References**

[1] J. G. Speer, D. V. Edmonds, F. C. Rizzo, and D. K. Matlock, Curr. Opin. Solid State Mater. Sci., 8, 219-237 (2004)

[2] A. J. S. T. Silva, H. Goldenstein, W. L. Guesser, M. F. Campos, Mat. Res., 17, 1115-1123 (2015)

[3] M. J. Santofimia, L. Zhao, and J. Sietsma, Metall. Mater. Trans. A, 40, 46-57 (2009)

[4] F. HajyAkbary, J. Sietsma, G. Miyamoto, T. Furuhara, and M. J. Santofimia, Acta Mater., 104, 72-83 (2016)

[5] G. Miyamoto, N. Takayama, and T. Furuhara, Scr. Mater., 60, 1113-1116 (2009)

Keywords: microstructure, high strength steel

Arthur Seiji Nishikawa (Department of Metallurgical and Materials Engineering, University of São Paulo, São Paulo, Brazil)

E-mail: arthur.nishikawa@usp.br

www.st-mat.imr.tohoku.ac.jp, pmt.usp.br/ltf