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Analytical TEM characterization of nanometric carbides in a microalloyed (HSLA) steel replica

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Abstract – Elemental analysis of fine precipitates within metal foils using transmission electron microscopy (TEM) can be highly problematic due to contributions from the matrix and its magnetic effects on microscope's alignment. The use of amorphous carbon extraction replicas circumvents most of these difficulties allowing the characterization of individual nanometric precipitates with complex structure and chemical composition (Fe, Mn, Cr, Mo, Nb, Ti, Al and C). Characterization of such precipitates is discussed.

The demand for steels with higher strength in the structural and pipeline industries has led to the development of high-strength microalloyed steels. Such steels usually contain low carbon levels to increase weldability, and are microalloyed with Ti, Nb, Mo or V, or a combination of these elements. Microalloyed carbides, nitrides and carbonitrides play a significant role in the control of austenite recrystallization and grain growth during steel processing and therefore leading to grain refinement [1]. The objective of this study was the analytical transmission electron microscopy (ATEM) characterization of nanometric precipitates of cementite and different types of carbides in a microalloyed steel replica.

The steel studied here was a low carbon microalloyed steel containing Fe (bal.), 0.28%C, 0.50%Mn, 1.06%Cr, 0.81%Mo and smaller additions of Ti, Nb and Al (wt%). Scanning transmission electron microscopy (STEM) and TEM analyses were performed at 200 kV using a JEOL JEM 2100F system equipped with a x-ray energy dispersive (EDS) Thermo Noran and electron (EELS) Gatan GIF Tridiem spectrometers. Morphological, chemical, size distribution and structural characterization were performed using conventional and advanced TEM techniques. Chemical composition for the different precipitates was analyzed through EDS. Crystalline structure of such carbides was studied using HRTEM, SADP in TEM and EDS spectral imaging (SI) through STEM analysis of carbides.

Microalloyed steel replica presented a high fraction of nanometric precipitates up to 300nm, mainly in the size range from 3 to 150nm. Very small precipitates, smaller than 20nm showed spherical shape, medium precipitates in the size range of 30 - 300nm showed elliptical, rectangular, elongated, and irregular shapes. Some micrometric Fe_3C precipitates appear as well, but in a reduced number and agglomerated in between nanometric precipitates. The precipitates can be described as: i) Cr doped cementite $(Cr,Fe)_3C$; ii) $M_{23}C_6$ carbides, composed by a combination of Cr with other elements such as Fe and Mo in different proportions; and iii) Some ultra-fine MC (few nanometers) precipitates, where there is the coexistence of Nb, Mo, and Ti, becoming (Nb,Mo,Ti)C.

Spectral imaging (SI) EDS showed in Fig. 1.b (in detail of Fig. 1.a - HAADF STEM image) allows the differentiation of $M_{23}C_6$ (right elongated) from MC (left spherical) nanometric carbides (Fig. 2). Detection and identification of fine precipitates (cementite and carbides) in microalloyed steel replica is a complex analysis and requires the use of a combination of advanced techniques in order to support the results. ATEM was employed successfully in the characterization and identification of different types of precipitates, such as very fine 5nm MC carbides in a microalloyed steel replica.

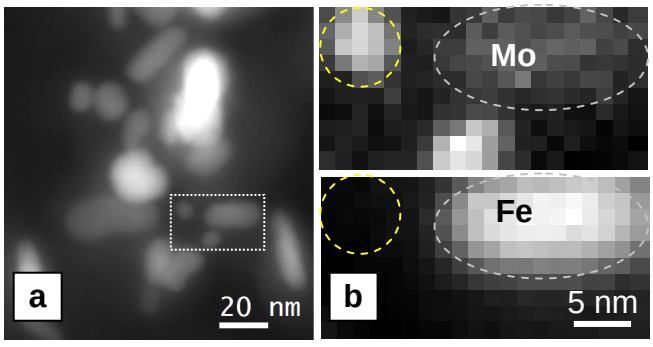

Figure 1: HAADF (high angular annular dark field) STEM image of very fine nanometric carbides in the microalloyed steel replica **a)** HAADF STEM **b)** Mo and Fe elemental maps from EDS SI.

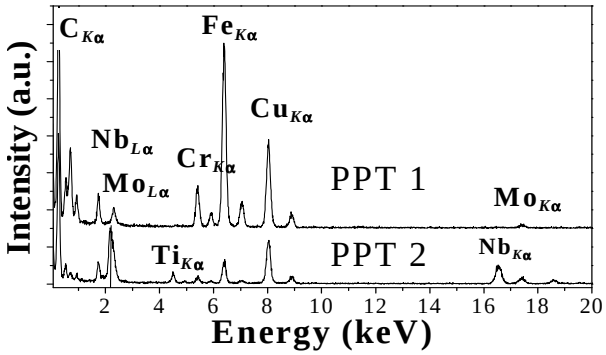

Figure 2: Energy dispersive spectra extracted from EDS SI data cube of $(Fe,Cr)_{23}C_6$ (right ellipsoid) from (Nb,Mo,Ti)C (left spherical) nanometric carbides showed in Fig 1.b.

References

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