

## Development of quantification methods applied to HSS alloys for carbides volume fraction and grain size assessments

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### Introduction

High speed steels (HSS) are used in applications where enhanced mechanical properties together with hot oxidation and wear resistances are required. Such an improved physico-chemical behaviour is mainly due to the presence of primary carbides within the microstructure. It is thus important to quantify the carbide volume fraction in HSS grades in order to set correlations between the microstructure and both the mechanical and the tribological properties of the materials. Depending of the initial chemical composition several types of carbides can precipitate in HSS such as MC (Nb and V-rich), M<sub>2</sub>C (Mo and W-rich), Cr-rich M<sub>7</sub>C<sub>3</sub>, etc. Two groups of HSS were analysed in this study, which are the so-called cast HSS grades that are manufactured by a centrifugal casting route and the cladded HSS manufactured by laser cladding. The metallurgical features such as carbides nature and shape have been determined that are related to the manufacturing process of the studied material, several methods have been used to quantify the carbide volume fraction and to assess the grain size.

### Materials and Methods

The studied materials are made of three cast HSS grades (AURORA, GALAXY and ZARYA), and three cladded HSS (A11, M4 and a mixed alloy made of 40%A11-60%M4). The carbides present in these materials are listed in Table 1. Their nature and shape have been determined in a previous work(1).

Table 1: Types of carbides observed in each studied material

Material	AURORA	GALAXY	ZARYA	A11	M4	40%A11-60%M4
Carbides	MC M <sub>2</sub> C	M <sub>7</sub> C <sub>3</sub> M <sub>2</sub> C	MC M <sub>2</sub> C	MC M <sub>7</sub> C <sub>3</sub>	M <sub>2</sub> C MC	MC M <sub>2</sub> C

A sequence consisting in a pre-treatment approach prior to image processing and a subsequent post-treatment is set in order to obtain the expected results. The pre-treatment approach is made of a specific metallurgical preparation of the sample followed by the use of an adequate microscope technique. All the techniques used to achieve the carbides quantification and the grain size assessment are summarized below:

- Sample preparation methods: as-polished, coloured/etched, and OPS (colloidal silica, which is an active oxide polishing suspension) conditions
- Microscopic techniques and analysis: optical microscope, scanning electron microscope (SEM) associated either with BSE (backscattered electron) or SE (secondary electron) detectors, EBSD (electron backscatter diffraction) combined with EDX (energy dispersive X-ray spectrometry)
- Image Post-treatments: with ImageJ, except for EBSD+EDX (Esprit software<sup>®</sup>)

### Results and Discussion

The different sequences that have been used to quantify the carbide volume fractions and to assess the grain size of the materials -are respectively summarized in Figure 1 and in Table 2.

#### **Carbides quantification**

The optical microscope (OM) is the preferred use for the carbides quantification, if there is enough



contrast between the phase that is analysed and the neighbouring matrix. Indeed, optical microscope allows a quick analysis, based on a large sampled area. Cast HSS AURORA is a good example of material analysed with optical microscope. MC carbides are quantified in the as-polished conditions while  $M_2C$  carbides are better enhanced after a colouring etching with Groesbeck reagent. Different post-treatments are applied on the rough optical images, as can be seen in Figure 2a for  $M_2C$  quantification, such as shading correction followed by various threshold algorithms available in ImageJ (we named this method Jcarbides). The actual MC and  $M_2C$  carbide amounts of AURORA grade fall within a narrow band which ranges between an upper and a lower limit respectively determined while using Shandbag and Li algorithms (2).

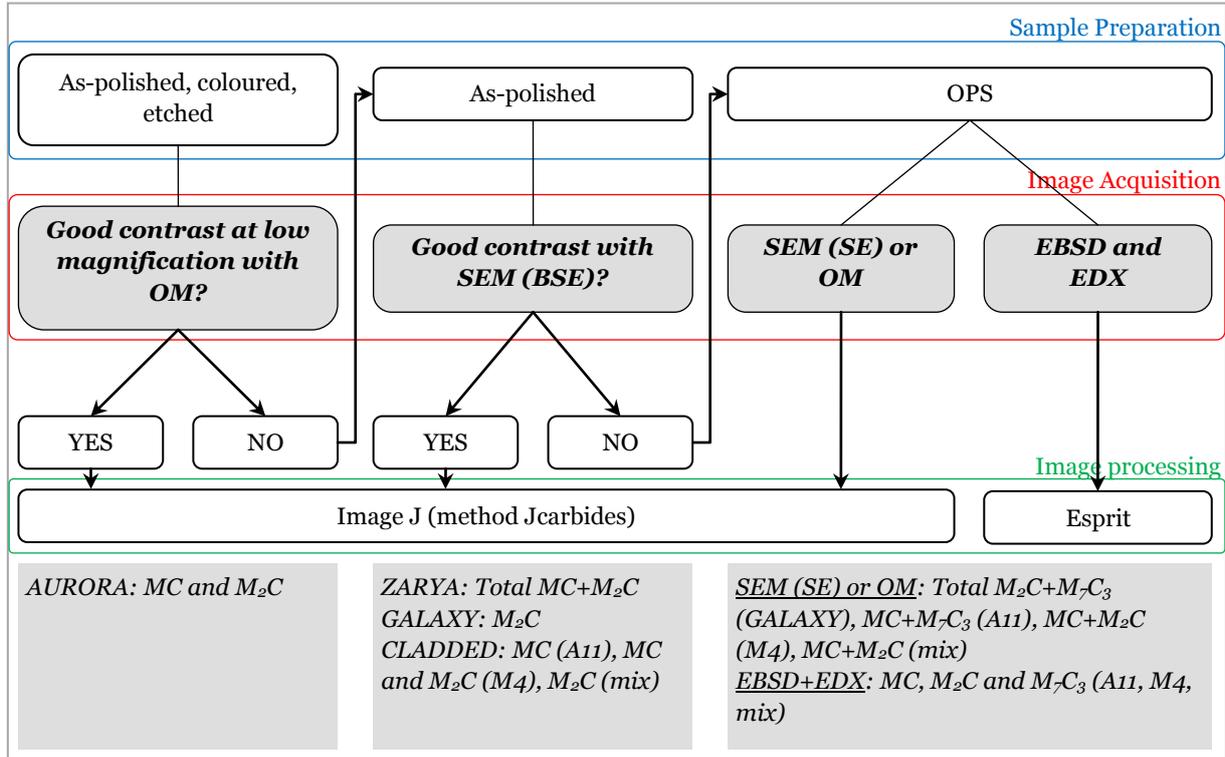


Figure 1: Carbides quantification methods depending on the studied material

Table 2: Grain size quantification methods depending on the studied material

Sample preparation		As-polished	Coloured Etched	OPS	
Microscopic technique and analysis		Optical microscope		SEM (SE)	EBSD+EDX
Material	AURORA				
	ZARYA, A11, 40%A11-60%M4				
	GALAXY				
	M4				
Image processing		Image J			Esprit

A similar approach based on optical microscope (OM) technique set up the limits to the use of such method for the other cast HSS grades. The reasons for the limitations are given below:

- ZARYA: thin morphology for both MC and  $M_2C$  carbides and insufficient contrast between these carbides and the matrix either in the as-polished or the etched conditions. Higher magnifications under optical microscope can be used at the expense of the sampled area that decrease down to a size which is no more representative of the studied material.
- GALAXY: Insufficient contrast between carbides and the neighbouring matrix in both as-polished and etched conditions (under colouring etching). Conventional etching with Villela reagent does not allow better results due to the large strip of matrix (retained austenite) in the vicinity of grain boundary carbides, which has the same light contrast as the unetched  $M_2C$  and  $M_7C_7$  carbides.

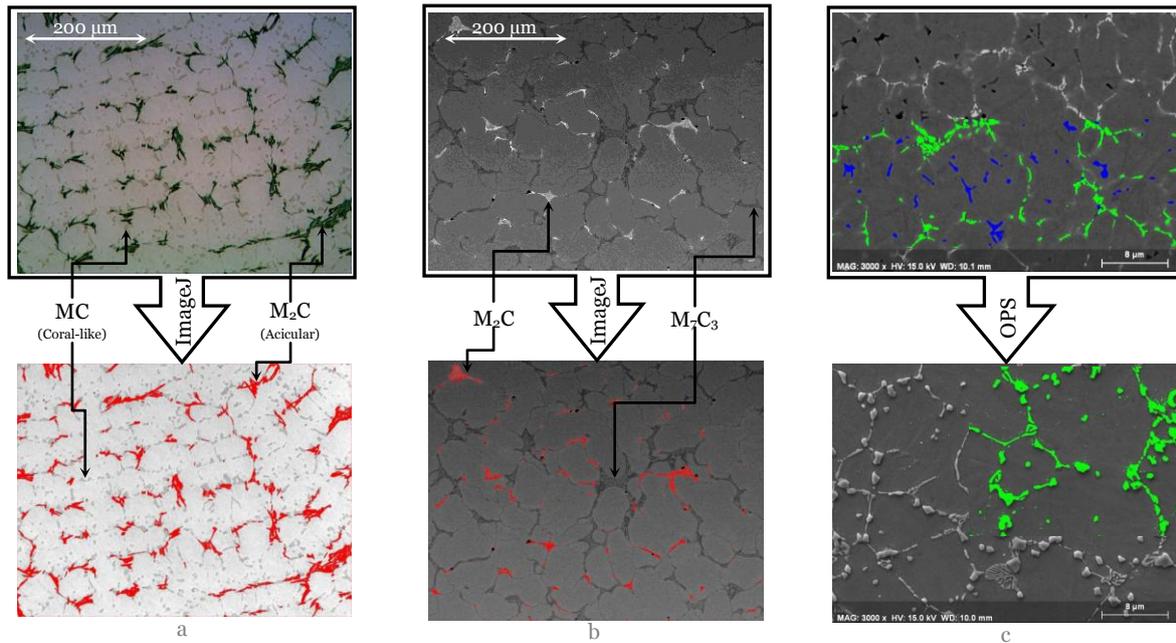


Figure 2: Carbide quantification using different sample preparation methods and microscopic techniques, post-treatment done with ImageJ – a:  $M_2C$  carbides quantification with OM technique and Groesbeck etching (AURORA); b:  $M_2C$  carbides quantification with SEM/BSE and as-polished conditions (GALAXY); c:  $M_2C$  (green) + MC (blue) quantification with SEM/SE and BSE, before and after OPS (M4)

The OM technique is not appropriate for the quantification of carbide phases in the cladded HSS due to their small size that is closed to the minimum optical resolution. Therefore the carbide content of cladded HSS was quantified in as-polished conditions with SEM/BSE mode (Figure 2c - top). With this approach a chemical contrast is obtained between carbides and the matrix due to the difference within the atomic mass of the elements entering in the composition of each phase. The lighter is the phase, the darker is the contrast. As the Fe-rich matrix appears in light grey contrast all the phases that contain elements with an atomic number either significantly lower (V-rich MC carbides) or significantly higher (Mo-rich  $M_2C$  carbides) than Fe can be quantified. Due to the small size of primary carbides present in the cladded HSS grades, only the upper limit for the carbides content is determined with the SEM/BSE technique. For the cast HSS ZARYA grade that contain both Nb-rich MC carbides and Mo-rich  $M_2C$  carbides, the SEM/BSE technique is also appropriate. The post-processing of SEM images is achieved while using a filter (median and mean) to remove the noise. Then an erosion operation is performed to obtain the lower limit of the primary carbide content, which allows taking over small particles corresponding to secondary carbides.

For the cast HSS GALAXY grade (Figure 2b) that contain both Cr-rich  $M_7C_3$  carbides and Mo-rich  $M_2C$  carbides the SEM/BSE technique performed in a similar way than that of ZARYA grade allows determining only the volume fraction of the later phase. The Cr-rich  $M_7C_3$  carbides cannot be quantified due to their grey contrast that is close to the light grey contrast of the neighbouring matrix. Similarly, such a limitation is also observed on Cr-rich  $M_7C_3$  carbides that are present in cladded HSS A11 grade, and on V/Mo-rich  $M_2C$  carbides found in the mixed cladded HSS A11/M4 grade. For the later carbides, a varying grey contrast that is close to the one of the matrix, and which originate from the chemical composition that also changes within a single carbide, represent the main limitation.

Nevertheless, OPS preparation makes it possible to determine the total amount of the primary carbide content for both cast HSS GALAXY grade (with OM technique) and cladded HSS grades as shown in Figure 2c - bottom (with SEM/SE technique). OPS preparation ran for several minutes enhances the roughness of carbides and so allows obtaining an improved contrast for these phases when compared to that of the matrix. The volume fraction of carbides obtained under such an approach is overestimated due to the topography between the carbides (not etched by OPS) and the matrix which

is partially dissolved by OPS. Hence a sufficient contrast is achieved between carbides and the matrix, the image processing is run in the same way like the method used with AURORA (method Jcarbides).

The carbide amount is also quantified under SEM/EBSD/EDX technique combining EBSD pattern with EDX spectra, and after an OPS sample preparation. Such a mixed approach allows determining the crystallographic structure of the phases (EBSD) and their chemical composition (EDX) and better results are obtained. Nevertheless, the analysed area must be small to obtain a good precision.

### **Grain size assessment**

The conventional approach used to determine the grain size is quoted within the standard ASTM E112. The most common way from this standard is the intercept method and such an approach has been used as the reference for the studied HSS materials with mixed results.

In fact, regardless of the sample preparation and the subsequent microscope technique, the conventional approach will yield a result whenever grain boundaries are well defined due to the presence of primary carbides describing a continuous network (cast HSS GALAXY and clad HSS M4). For grades having a semi-continuous network of primary carbides at grain boundaries the classical approach leads to unsatisfactory results as the grain size obtained seems to be higher than the actual one (cast HSS AURORA). This conventional approach could not be performed when the grain boundaries are not fully defined by carbides (cast ZARYA, clad A11 and mixed A11/M4).

Therefore, the EBSD technique performed under the SEM seems to be more appropriate to fill the gaps observed within the previous technique. In addition, the EBSD technique applied to samples where the conventional approach was performed seems to give more relevant results for the grain size assessment. In fact the criteria used to distinguish adjacent grains under EBSD, which is based on a minimum disorientation value between their main lattice parameters, is more relevant to better understand the mechanical behaviour of the material at the grain scale. Moreover, when the matrix is made of martensite with packets of laths having different orientation inside a single prior austenitic grain the EBSD approach is more efficient to predict the bulk material behaviour (macro scale) while allowing a highlight of the sub grains that set the behaviour at the micro scale. However, as for the carbides quantification, the studied areas in EBSD are very small and so the assessment with conventional approach where large regions of interest can be considered remain of interest in order to obtain a first approximation of the grain size.

## Conclusions

Different methods were tested to quantify the carbides volume fraction and to obtain the grain size on different HSS materials.

The carbide volume fraction can be obtained under the following approaches:

- OM technique with as-polished or etched samples if the contrast between the carbides and the matrix is high enough and if the carbides are large in size;
- SEM/BSE technique with as-polished samples for carbides containing both heavy and light elements;
- OM or SEM/SE techniques with OPS samples to obtain the total carbide phase fraction.

The grain size can be assessed while using the following approaches:

- OM or SEM techniques if the grain boundaries are well defined;
- SEM/EBSD+EDX technique if not, and also for enhancement of sub grains size.

## References

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