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Combined EBSD, SEM and EDS for detecting minor phases on austenitic stainless steels

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ABSTRACT

Phases in AISI 347 austenitic stainless steel under heat treatments simulating post welding and aging effects are characterized. The study is performed with a scanning electron microscope by using electron backscattered diffraction, secondary and backscattered images and X-ray maps. The potential of each signal is analyzed for the identification and quantification of austenite, ferrite, carbides, silicates, sulfides, and sigma phases. Since the sigma phase and the chromium carbide are difficult to identify by conventional analysis, the method of Mean Shift Clustering has been implemented to face this issue. The results evidence that this approach complements the ease and availability characterizing EDS analysis and the capability of EBSD for crystallographic phase identification.

1. Introduction

Austenitic steel weld deposits on carbon or low alloy steels are commonly chosen for building pressure vessels intended to preserve the base material from corrosion [1]. In the field of nuclear industry, the cladding of reactor pressure vessels aims also to reduce the ion transport that can be activated when crossing the reactor nucleus, consequently lowering the collective installation dose. In the design of alloys a balance between elements favoring the appearance of δ -ferrite and γ -austenite must be considered [2,3]. Elements such as molybdenum, nitrogen, carbon, silicon and, in the case of stabilized steels, titanium and niobium are also responsible for the formation of secondary phases such as carbides, nitrides, sulfides, silicates and intermetallic phases; some of them are detrimental to the material performance [4,5].

The presence of the ferrite phase turns relevant to avoid the hot cracking phenomenon scanning during solidification [2]. This phase becomes unstable at post welding heat treatment temperatures, and may result in several minor phases. One of them is the sigma phase (σ), both affecting corrosion properties and also reducing material toughness [6,7]. The latter effect must be particularly borne in mind when prolonged facility active life is expected, since safe and reliable operation must be ensured. For these reasons, the characterization of precipitation mechanisms and the evolution of this phase during service is critical. For

a better comprehension of such phase transformations, a thorough microstructure characterization of both as-cast and aged alloys is important.

Scanning electron microscopy (SEM) has become a versatile tool frequently used in materials science, involving different complementary techniques, which provide structural, compositional and morphological information. Specimen topographies may be surveyed by secondary electron imaging (SEI), whereas chemical contrast distributions can be achieved through backscattered electron images (BEI). In addition, with the aid of an energy dispersive spectrometer (EDS), it is possible to perform quantitative elemental analysis, as well as to acquire X ray maps (XRMs) in order to provide elemental spatial distributions [8]. All these techniques are relatively fast, especially when a silicon drift detector is available, which allows to further reduce acquisition times. Another complementary technique, which has gained relevance during the last decades, is electron backscatter diffraction (EBSD), permitting to survey crystallographic information at submicrometer scale [9,10]. If all these data are carefully processed and gathered by means of a meticulous statistical analysis, as multivariate methods, a thorough characterization of the specimen under study may be achieved.

In this work, different heat treatments simulating post welding and service conditions were applied for AISI 347 steels, commonly used in the nuclear industry. These samples were characterized in an SEM

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taking advantage of the conventional BEI, SEI and EDS signals, as well as EBSD. The minor phase identification performances through the different images are compared, and a strategy combining the analyses by means of these various signals is presented, which enables to univocally distinguish the phases present on these samples.

2. Experimental

The nominal composition of the AISI 347 austenitic stainless steel samples studied is (in wt%) C: 0.03–0.04, N: 0.021–0.023, Si: 0.94, P: 0.017, S: 0.006, Ti: 0.012, Cr: 18.9–19.0, Mn: 0.97–1.00, Co: 0.03, Ni: 11.2–11.3, Nb: 0.567–0.57, rest Fe. In order to achieve the ideal temperature and time values representing service conditions, an Arrhenius extrapolation was carried out. For a service temperature T_s and a period t_s , the equivalent laboratory temperature T_L and time t_L can be estimated from [11,12]:

$$t_L = t_S exp\left[\frac{Q}{R}\left(\frac{1}{T_L} - \frac{1}{T_S}\right)\right]$$
(1)

where *R* is the ideal gas constant and *Q* the activation energy. For a reactor operating at 300 °C during 30 years and expectation to prolong up to 60 years, eq. (1) predicts a laboratory treatment at 452 °C during 285 h (aging), if the activation energy is 200 kJ/mol [13]. The post welding heat treatment (PWHT) is attained by heating the sample at 600 °C for 40 h. The PWHT settings used in each case are summarized in Table 1. The sample named AW corresponds to "as welded", i.e., with no PWHT.

After applying heat treatments, samples were embedded in PolyFast Struers resin, sanded at 180 rpm with 1200 μ m paper during 1 min, successively polished at 150 rpm with 9, 6, 3 and 1 μ m diamond pastes along 5, 4, 4 and 3 min, respectively, and finished with colloidal silica (0.05 μ m) at 120 rpm for 10 min. The force applied was 18 N in all cases.

All measurements were performed in a Σ igma-Carl Zeiss FE-SEM with a Schottky electron gun, equipped with an Oxford Nordlys Nano EBSD detector with a CCD camera (1344 \times 1024 pixels) and a front phosphor screen (40 mm \times 35 mm), and an SDD X-ray detector (80 mm² front area) with a nominal resolution of 127 eV @Mn-Ka. An incident beam energy of 20 keV was used for all images and X-ray Maps.

EBSD measurement settings were optimized in a previous work [14]: 20 keV beam energy; 8.5 mm working distance; 120 μ m aperture; 70° tilt angle (angle between incident beam and the direction normal to the sample surface ND); 0.05, 0.1 and 0.2 μ m step sizes; and 4 \times 4 binning for the CCD camera. Crystallographic data included in the EBSD acquisition software were used to identify all phases, except the sigma phase, for which crystallographic data given in [15] were used. EBSD maps were processed with Channel 5 software (Oxford Instruments): wild spikes were eliminated and then non-indexed points were extrapolated using 8, 7, 6 and 5 neighbors. Cleaning routines, in general, removed <5% of the non-indexed points.

3. Results and discussion

3.1. Austenite and ferrite

The major (austenite) and second (ferrite) phases can be easily detected and quantified by conventional SEI and BEI (see Fig. 1 a and b). These phases can be distinguished in SEI by the small relief generated by

 Table 1

 Heat treatments applied to AISI 347 austenitic steels studied in this work.

Sample PWHT at 600 °C		Aging at 452 $^\circ\mathrm{C}$	
AW	_	-	
40 h	40 h	_	
100 h	100 h	_	
PWHT+A	40 h	284.4 h	

polishing [16]: ferrite is more resistant to polish than the austenite matrix in stainless steels [17]. In BEI, depending on the experimental conditions, a contrast in the mean atomic number of around 0.1% can be detected [18]. Bearing this in mind, ferrite-austenite should be easily detected by BEI, since their density contrast is around 2.5–3%. In XRMs, a lesser/higher amount of Nickel/Chromium in ferrite/austenite is clearly observed (Fig. 1 c and d) and can also be used to assign these phases.

On the other hand, EBSD properly identifies these two phases due to their different crystalline structure (ferrite bears a BCC crystal structure, while austenite is FCC). EBSD maps give additional information about the crystalline orientation: ferrite grows principally in austenite grain boundaries, exhibiting an elongated shape (around 1 µm wide); whereas austenite is highly oriented in samples under heat treatments (40 h, PWTH+A, 100 h), with a preferential orientation resulting from the alignment of the {100} planes with the direction normal to the sample surface ND. It can also be observed that the ferrite/austenite (δ/γ) boundaries satisfy the Kurdjumov-Sachs (KS) [19] relationship: $\langle 110 \rangle_{\gamma} / /$ $\langle 111 \rangle_{\delta}$ and $\{111\}_{\nu}//\{110\}_{\delta}$ (see Fig. 2), which was previously reported by Monlevade et al. [20] for duplex stainless steels and by Singhal et al. [21] for austenitic steels. Sample AW satisfies the KS relationship, whereas in the case of ferrite, PWHT+A presents another preferential orientation which is around 10° rotated with respect to KS, and sample 100 h exhibits a non-homogeneous orientation distribution.

3.2. Niobium carbide

NbC is present in all samples. It can be clearly observed in BEI due to its higher mean atomic number, as compared with other phases present (Fig. 3). The main growing sites of NbC are γ/γ and γ/δ grain boundaries.

Since NbC has the same crystalline structure as austenite, its appropriate indexation in EBSD maps is not straightforward. Correct indexation was possible in a few cases in which the NbC phase size was >2.5 \times 2.5 μm^2 and the polishing extent was good enough to produce high quality Kikuchi patterns. One of these cases is shown in Fig. 4, where it can be observed that some points corresponding to NbC are satisfactorily indexed, although there are some pixels erroneously assigned to austenite or not indexed. EBSD in combination with X-Ray maps are useful to perform a correct phase identification, minimizing the mis-indexation problems. According to the EBSD measurements, the orientation relationship between austenite and NbC is $\{001\}_{\gamma/}/\{001\}_{NbC}$, as previously observed by other authors [22,23] (Fig. 5).

3.3. Silicates and sulfides

Silicates and sulfides could not be detected by EBSD measurements in this work. These phases did not allow Kikuchi patterns, which can be associated with insufficient polishing or to the presence of vitreous phases. Since EBSD does not give information in this case, its detection must be performed by other techniques. SEM images allow the detection of precipitates by observing the circular dark zones (Fig. 3). Nevertheless, the assignment of the precipitate type should be carried out by EDS analysis.

Both silicates and sulfides were observed in all samples; although they could be qualitatively distinguished, their chemical composition could not be accurately determined. This could be explained bearing in mind that in most cases both precipitates grow together in a region of around $\sim 1 \ \mu m^2$, which is smaller than the EDS spatial resolution [14]. By observing XRMs, it can be inferred that the precipitates are mainly manganese silicates, manganese sulfides and, to a lower extent, iron silicates. Kim et al. [24] and Lee [25] reported the joint precipitation of Mn-silicates and MnS, which was also observed in the samples studied along the present work (Fig. 6). In addition, the presence of these precipitates was observed near NbC and, in some cases, with other Ti- and Cr- rich phases (Fig. 6).



Fig. 1. SEI (a), BEI (b), Cr-Ka (c) and Ni-Ka (d) XRMs corresponding to sample 100 h. Arrows indicate ferrite phase.

3.4. Sigma

The σ -phase is one of the most interesting phases to detect and characterize in austenitic steels, since it highly affects corrosion resistance and material toughness. It is a Cr-rich phase with tetragonal lattice [11], its precipitation mainly occurring due to the diffusion of Cr. Other factors affect the σ -phase formation; in general, all ferrite forming elements like Cr, Nb, Ti, Mo or W promote precipitation of σ -phase [6,26], but due to differences in the diffusion rate of the alloying elements, the formation of σ -phase in austenite is about 100 times slower than in ferrite. Consequently, ferrite-rich regions in austenitic alloys become beneficial for σ -phase precipitation [27,28].

The unequivocal detection of the sigma phase turned out to be almost impossible by SEI, and very difficult and sometimes ambiguous by BEI and XRM. This is mainly because the chemical compositions of sigma phase and ferrite are similar. In addition, the sigma phase, when present, is expected to occur in very low amounts and located near ferrite, as stated above. Since the crystalline structure of the sigma phase is different from those corresponding to the other phases present, EBSD may help to univocally detect it as long as correct polishing conditions are achieved. Sigma phase was found in samples under long PWHT (100*h*) and aging treatments (PWHT+A); this is exemplified in Fig. 7, where EBSD maps for both samples are shown.

The sigma phase was observed to precipitate in ferrite-austenite grain boundaries, and to follow the crystallographic relationship given by $\{111\}_{\gamma}//\{001\}_{\sigma}$ and $\langle011\rangle_{\gamma}//(140\rangle_{\sigma}$ [29,30] (Fig. 8), in some cases along with chromium and niobium carbides. Singhal et al. [21] and Barcik [27] have explained this feature, pointing out that the growth of the sigma phase occurs at expense of chromium carbide. In stainless steels, the classification of the morphology of the σ -phase can be divided into four types [31]: 1) grain boundary precipitation, 2) triple point precipitation, 3) corner precipitation and 4) cellular precipitation. Along this work, mainly σ -phase type 1 was observed.

The mean grain size for sigma phase resulted in $(0.41 \pm 0.05) \text{ mm}^2$ and $(0.45 \pm 0.01) \text{ mm}^2$ for sample PWHT+A and 100 h, respectively. It is important to determine the grain size by performing some EBSD maps at different step sizes. Once an idea of the mean grain size is obtained, the subsequent EBSD maps can be optimized to ensure at least 3 points measured in minor phases (for instance, in the case of quantification or crystallographic analysis).

3.5. Chromium carbide

Chromium carbide was observed in samples PWHT+A and 100 h, which also exhibit sigma phase. Since chromium carbide and austenite have the same crystalline structure [23], SEM-EDS complements EBSD, allowing to discriminate regions with high Cr content (associated to chromium carbide). However, EDS alone will not suffice to unambiguously discriminate between sigma phase and chromium carbide. Fig. 9 a) shows EBSD results, where only austenite and ferrite were detected and indexed, whereas chromium carbide is not distinguished from the matrix. Nevertheless, the Cr XRM exhibited in Fig. 9 b) displays regions with high Cr content; in addition, these regions do not follow the crystallographic orientation for austenite, as evidenced in Fig. 9 c). This example demonstrates that these regions must be unequivocally reassigned as CrC, as shown in Fig. 9 d). Furthermore, regions not indexed were identified according to the XRMs as silicates, sulfides and NbC. The re-assignation of the other phases not indexed by EBSD or mis-indexed can be performed manually, by associating the zones with high content of an element with the corresponding phase, or by means of an algorithm for phase clustering (see below).

The very low amount of chromium carbide limits the ability to furnish pole figures. As a consequence of the low statistics corresponding to this case, a reliable assessment of the precise orientation relationship is unattainable, and the phase amount estimation bears high uncertainties. Despite these difficulties, it is possible to conclude that



Fig. 2. Pole figures for γ -austenite (•) and δ -ferrite (•) phases. The central axis coincides with ND. 1: $\langle 110 \rangle_{\gamma}$ and $\langle 111 \rangle_{\delta}$; 2: $\{111\}_{\gamma}$ and $\{110\}_{\delta}$; 3: $\{100\}_{\gamma}$. Contours in pole figures correspond to higher density of population.

chromium carbide was only observed in samples under long heat treatments, located at the interface δ/γ , with the $\langle 111 \rangle$ or $\langle 011 \rangle$ directions parallel to ND.

3.6. Mean shift clustering

Up to now, in order to identify the different phases present in the samples under study, the ability of the different signals produced in an SEM has been brought to evidence. Several features (composition, morphology, crystallographic relationships, grain structure, etc.) can be characterized in a non-destructive way by means of the various images acquired. For the identification of some phases, EBSD maps have been strictly necessary, as is the case of the sigma phase. Since these maps are rather time consuming (considering sample preparation and measurement) and usually EBSD detectors are not available in a conventional SEM, a question naturally arises: is it possible to discriminate and quantify all phases avoiding this technique? In this sense, other signals provided by an SEM can be further explored to take the maximum advantage and extract all the pursued information.

Multivariate statistical analysis methods can become very useful to this aim. For instance, Principal Component Analysis [32] and



Fig. 3. BEI for sample 40 h. The lighter phase (pointed with arrows) corresponds to the high Z rich phase (NbC in this case). Dark rounded regions are precipitates.



Fig. 4. Nb X-ray (left) and EBSD (right) maps for sample 100 h. •: ferrite, •: NbC, •: FCC matrix, ●: not indexed.

Multivariate Curve Resolution [33] have been successfully applied to materials characterization by XRMs. More recently, Schmaedech et al. [34] implemented Mean shift clustering (MSC) to identify phases through XRMs originating in mineral samples, which proved to be more efficient than other image-segmentation methods. On this basis, MSC has been chosen to analyze XRMs acquired for the austenitic stainless steel samples under study.

In a few words, the strategy followed in MSC is to find data clusters in multivariate datasets. In the present case, these datasets are furnished with XRMs, which allow to associate each surface pixel with a vector whose components are the x-ray characteristic intensities for each atomic element detected. These vectors are expected to constitute clusters with similar features, which should correspond to pixels with similar concentrations. In order to define these clusters, an iterative evolution (shifting) of a kernel towards the average of the data points involved is carried out; this evolution is governed by a tolerance parameter representing a multidimensional radius, called bandwidth. The kernel will stop moving after a number of mean shifts, when a maximum of the density of data points is found. In this cluster center, all



Fig. 5. Pole figures for austenite (\bullet) and NbC (\bullet) phases for sample PWHT+A. The central axis coincides with the direction perpendicular to the sample surface. 1: {001}_{γ} and {001}_{NbC}; 2: {011}_{γ} and {011}_{NbC}; 3: {111}_{γ} and {111}_{NbC}.



Fig. 6. SEI of precipitates for samples AW (a), 100 h (b) and PWHT+A (c). Silicates, sulfides, NbC and an unknown phase with high Ti and Cr content, inferred from XRMs are indicated. XRMs corresponding to image (c) are also shown.

members have similar features; this procedure is repeated until each data point is associated with a cluster. The choice of the bandwidth is initially arbitrary; a good estimate may be achieved by starting with a small value, which may artificially result in too many different phases, and after carefully increasing this value, the number of phases is reduced and should stabilize in a realistic value (see ref. [34] for more details).



Fig. 7. Raw EBSD phase maps for the samples presenting sigma phase PWHT+A (a,b) and 100 h (c,d). •: sigma phase, •: ferrite, •: FCC matrix, •: not indexed.

The XRMs which can be considered for applying MSC must be those offering reliable statistical uncertainties, enabling phase discrimination in the regions of interest. In the present study, major components (Cr, Fe and Ni) were always used, and in some particular cases, Si, Nb and Mo could also be included, as indicated in each legend of the examples discussed below.

In order to test the ability of this approach for identifying phases in austenitic stainless steels, it was applied to the XRMs acquired in samples 100 h and PWHT+A. Fig. 10 shows an example in which EBSD maps allowed to identify only three phases, and many pixels could not be indexed. MSC was applied to the relevant XRMs (Cr, Fe Ni and Si), resulting in four different phases, which can be assigned to γ, δ, σ , and a Si-rich phase. It can be seen that all pixels not indexed by EBSD have been attributed to different phases by MSC, mainly identifying those in γ - δ grain boundary pixels as sigma phase.

It can be seen in the example shown in Fig. 11 for sample PWHT+A, that the NbC phase mis-indexed by EBSD has been properly identified by MSC. The spatial distribution of sigma and ferrite phases does not exactly match, although certain correlation can be established between both techniques. This discrepancy may be attributed to the spatial resolution associated with each signal, since the interaction volume involved in X-ray generation (a few μm^3 [35]) is much larger than the region originating the Kikuchi patterns, which embraces a few nm^3 [14]. The EDS signal spreading beyond the irradiated area may cause the identification of phases greater than their real dimensions, or even reveal false phases. This different spatial resolution from each signal is



Fig. 8. Pole figure showing austenite/sigma γ/σ orientation relationship for sample PWHT+A. The central axis coincides with ND. a) Pole figures for austenite (\bullet) and sigma (\bullet) phases for {111}_y-{001}_{σ} (1) and $\langle 011 \rangle_{\gamma}$ -{140}_{σ} (2); b) crystal orientation of γ and σ unit cells is shown.



Fig. 9. Identification of chromium carbide for sample PWHT+A by EBSD maps and XRM. a) EBSD phase indexation without EDS, b) XRM corresponding to Cr, c) Inverse pole figure (IPF X), d) EBSD+EDS indexation. •: ferrite, •: silicates, •: FCC matrix, •: NbC, •: CrC, •: sulfides, •: not indexed (EBSD).

also reflected in Fig. 12, where the region associated to the sigma phase according to MSC is larger than that identified by EBSD. In addition, MSC is capable of discriminating two phases with high Cr content: one of them can be assigned to CrC with lower Fe content, and the other one corresponds to a sigma phase —the identification of CrC by EBSD becomes complicated, due to the similarities in the crystallographic parameters with the γ matrix. These results imply that it must be taken into account that the signal on which each technique is based reveals features originating in different interaction volumes, providing complementary information: EBSD evidences the crystallography of a few surface atomic layers, whereas XRMs provide elemental composition from greater

sample depths.

Fig. 13 displays the phase distribution maps obtained by MSC and EBSD for another region in sample PWHT+A. It is clear that only two phases are indexed by EBSD (prior to the post-processing treatment), whereas the MSC routine implemented distinguishes five different phases, including phases with higher Si and Nb content, which are clearly discriminated.

3.7. Phase amount estimation

Phase concentration was estimated from the acquired images. As previously mentioned, chromium carbide was detected in samples PWHT+A and 100 h, but could not be quantified due to the low amount present in the samples. The results obtained are displayed in Table 2. Uncertainties in austenite concentration run between 0.2 and 0.5%, whereas for minor phases the uncertainties are around 10% for ferrite, 7–19% for NbC, 18–24% for silicates, 20–53% for sulfides and 14–33% for sigma.

Elements constituting carbides, as Cr and Nb, may trap carbon atoms, decreasing the amount of this element in the matrix, which modifies the sensitization of the material under consideration [36]. Considering the uncertainties involved, the silicate and sulfide contents do not bear noticeable variations, except for a slight decrease in the sample under the largest heat treatment at the highest temperature (100 h). For the samples presenting sigma phase, it can be seen that increasing the time for the heat treatment at 600 °C favors the nucleation of new sigma phase grains more than the growth of the existing ones. Such growth of sigma grains is expected for higher temperatures or heating times. It must be borne in mind that the reduction of toughness and ductility associated with the presence of σ phase has been repeatedly studied [37,38], and is often referred to as " σ phase embrittlement" [2].

4. Conclusions

In this work we considered the problem of achieving an adequate and precise description of phases present in AISI 347 steel by the information obtained in an SEM: EBSD, BEI, SEI, XRM. Samples were submitted to different heat treatments, simulating post welding and nuclear reactor operation conditions.



Fig. 10. Left: Cr, Ni, Fe and Si XRMs from sample 100 h; center: image produced by MSC; right: EBSD. •: sigma phase, •: ferrite, •: silicate, •: FCC matrix, •: not indexed (EBSD).



Fig. 11. Left: Cr, Nb, Fe and Ni XRMs from sample PWHT+A; center: image produced by MSC; right: EBSD. •: sigma phase, •: ferrite, •: NbC, •: FCC matrix, •: not indexed (EBSD).



Fig. 12. Top: Cr, Fe and Ni XRMs from sample PWHT+A; bottom, left: image produced by MSC; bottom, right: EBSD. ●: sigma phase, ●: ferrite, ●: NbC, ●: CrC, ●: FCC matrix, ●: not indexed (EBSD).

In the stainless steel studied, the austenite matrix was highly oriented with its {100} planes in the direction normal to the sample surface. Heat treatments enhance this orientation. Ferrite was found to mainly grow at the austenite grain boundary, and, to a lesser extent, inside the austenite matrix. The relative ferrite/austenite orientations satisfy the Kurdjumov-Sachs relationship.

Niobium carbide was observed in all the samples studied. It grows in the austenite/austenite and ferrite/austenite grain boundaries and it is oriented similarly to austenite. Silicates and sulfides appeared to preferentially precipitate together, although they were also found separately, in the vicinity of niobium carbides. Other Ti and Cr rich phases were observed, but their elemental composition could not be clearly determined.

Sigma phase was univocally identified with EBSD. The procedures PWHT and PWHT+aging lead to the formation of this phase, which precipitates in the austenite-ferrite grain boundary with an orientation relationship given by $\{111\}_{\gamma}//\{001\}_{\sigma}$ and $\langle011\rangle_{\gamma}//(140)_{\sigma}$. The mean

grain size of the sigma phase is indistinguishable for the two samples exhibiting this phase, which means that the heat treatments applied influence more in grain nucleation than in grain growth. Samples presenting sigma phase also bear chromium carbide with the $\langle 111 \rangle$ or $\langle 011 \rangle$ direction parallel to ND.

Clearly EBSD measurements involve large acquisition times, and this technique is not commonly available in most SEMs; in this sense, it is worth devoting most effort to the thorough processing of the conventional signals (SEI, BEI and XRMs) in order to discriminate minor phases, and use EBSD as a complementary tool for microtexture analysis. On the other hand, MSC must be used specifically to identify and quantify phase compositions, a successful routine requiring an adequate level of statistics. Care must be taken when complementing the information provided by both techniques, since their spatial resolutions are orders of magnitude different, which may hamper the phase identification procedure.

The present explorative study on the MSC procedure for phase



Fig. 13. Top: Cr, Fe, Mo, Nb, Ni and Si XRMs from sample PWHT+A; bottom, left: image produced by MSC; bottom, right: EBSD. •: ferrite, •: silicates, •: FCC matrix, •: NbC, •: CrC, •: sulfides, •: not indexed.

Table 2

Percentage phase concentration estimated by SEM images for the samples studied in this work.

Sample	Austenite	Ferrite	NbC	Silicates	Sulfides	Sigma
AW	93.4 ± 0.5	3.8 ± 0.3	$\begin{array}{c} 0.25 \\ \pm \ 0.04 \end{array}$	$\begin{array}{c} 0.38 \pm \\ 0.09 \end{array}$	0.17 ± 0.09	-
40 h	96 ± 2	2.5 ± 0.4	0.4 ± 0.2	$\textbf{0.7}\pm\textbf{0.4}$	$\begin{array}{c} 0.05 \pm \\ 0.03 \end{array}$	-
100 h	$\begin{array}{c} 96.9 \pm \\ 0.2 \end{array}$	3.3 ± 0.3	$\begin{array}{c} 0.28 \\ \pm \ 0.02 \end{array}$	$\begin{array}{c} \textbf{0.29} \pm \\ \textbf{0.07} \end{array}$	$\begin{array}{c} 0.04 \pm \\ 0.02 \end{array}$	$\begin{array}{c} \textbf{0.27} \pm \\ \textbf{0.09} \end{array}$
PWHT+A	$\begin{array}{c} 96.2 \pm \\ 0.3 \end{array}$	$\begin{array}{c} 2.9 \ \pm \\ 0.3 \end{array}$	$\begin{array}{c} 0.37 \\ \pm \ 0.07 \end{array}$	$\begin{array}{c} \textbf{0.40} \pm \\ \textbf{0.07} \end{array}$	$\begin{array}{c} 0.05 \pm \\ 0.01 \end{array}$	$\begin{array}{c} \textbf{0.07} \pm \\ \textbf{0.01} \end{array}$

identification in this kind of steels demonstrates the potentialities of this approach. A promising further step may involve additional signals, such as concentration maps instead of 255 level XRMs, to better determine the phase distribution and the average element concentration in each phase. Additionally, MSC may be useful to reveal regions in which phases are emerging; for instance, high Cr content ferrite which may evidence the nucleation of sigma phase.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request. The raw/processed data required to reproduce these findings cannot be shared at this time due to technical or time limitations.

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S.P. Fernandez Bordín et al.

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