Experimental report

07/10/2016

Proposal: 1-03-31 **Council:** 4/2015

Title: Characterization of Roman welding technique

Research area: Materials

This proposal is a new proposal

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Samples: Roman Iron

Instrument	Requested days	Allocated days	From	To
D1B	2	0		
D20	1	2	11/07/2016	13/07/2016

Abstract:

The proposed experiment aims to study this Roman welding technique. Previous results have shown that the welding is characterized by a large contact surface between two joined bars of possible different Carbon contents on large contact areas and probably at high temperature (1100°C-1200°C). The objective is to characterize the two sides of the full length joining in order to get information on phase qualification and Carbon content and the texture. The texture measurement will help to understand the condition of hammering. With respect to these ancient and very specific materials, non-invasive techniques are required for solving these points. For this reason, neutrons diffraction is adequate solution.

Identification of welding in roman bars from shipwrecks archaeological discovery (Saintes Maries de la Mer, France)

Aims of the experiment and scientific background

The proposed experiment aims to study the Roman welding technique from iron long quadrangular bars (1L type) coming from well identified shipwrecks (-100/ +100 JC) recently discovered in Mediterranean sea (south of France, Saintes Maries de la Mer). The objective was to characterize the two sides of the full length weld in order:

(i) to get information on phase qualification and Carbon content of the weld and of the two welded iron parts;

(ii) to evaluate the quality techniques of welding on iron bars, based on non-destructive approach.

Experimental results:

1. Experimental conditions:

Two quadrangular fragments of bars were analyzed: (1) milled and polished SM9 (L3) sample and (2) rough corroded SM2-2 (L3) sample. Both fragments exhibit a large weld with S shape, of about 10 cm length (fig. 1).

Samples were put vertically on the X-Z automatic control holder, positioning the S weld surface perpendicularly to the neutrons beam. The X axis was corresponding to the thickness of the bar segment (~ 2 cm) and the Y axis was corresponding to the length (vertical position). Linescans were performed at different Y values along the X axis. Around 14 to 16 points were recorded on each X linescan. This is schematized in Fig. 1 for SM9. Two batches of analysis and a standard calibration were performed. All the experiments were performed at λ=1.12A. Experimental setup is summarized hereafter (Table 1).

	Beam slots*	**Sample**	**Y linescan mm* (u.a.)**	**X scan step****	**Acquisition time / point**	**Files number**
Batch 1 (Z distance = iron depth within incident beam neutrons ~25 mm						
	Beam size ~X=1 x Y=3 mm²)	Roman Iron bar SM9 (L3)	166 / 126 / 98 / 71 / 41 / 07	2mm	360 s (except for 166 mm : 240 s)	SM9-950525 up to SM9-950532
		Roman Iron Bar SM2-2 (L3)	128 / 98 / 88 / 78 / 68 / 58 / 48 / 38 / 08 +148	2mm	360 s	SM2-950534 up to SM2-950543
Standard calibration						
	X=1mm Y=3mm	Rod 9mm diam.	Na2Ca3Al2F14	No scan	600 s	950544 up to 950548
	X=12 mm Y=8 mm	*idem*	*idem*	*Idem*	*idem*	950549
Batch 2 (Z distance = iron depth within incident beam neutrons ~ 8 mm)						
(vertical scan at X~middle of the bar section)	X=12 mm Y=8 mm (beam size ~10x3mm²)	SM9 (L3)	126 / 98 / 71 / 41 / 07 (milieu de la section)	Large beam (beam size ~X=10mm, Y=3 mm)	900 s	950552 up to 950556
	X=2mm Y =5mm (beam size ~X=1 x Y=3 mm²)	SM9 (L3)	98 (15 pts) / 41 (15 pts)	1.5 mm	1800 s	950559 & 950560

Table 1. Experimental setup – iron bars neutron diffraction. * The origin is here arbitrarily defined. ** This is corresponding to around 14 to 16 measured points performed for each Z linescan.

2. Evidence of different phases within quadrangular bars

Diffraction results show that the main phase identified is alpha iron (ferrite). Another minor phases was also observed (Figure 2). But no clear variation of internal variation on either side of the weld was possible due to the high background of the diffraction signals. It was found that a variation linked to the weld can be observed for both iron bars tested but the weld remains quite finely located inducing any a very thin perturbation in the section.

Thus to this very positive diffraction information, the experimental conditions were improved and adapted to both increase the S/N ratio and to focalize information on specific areas.

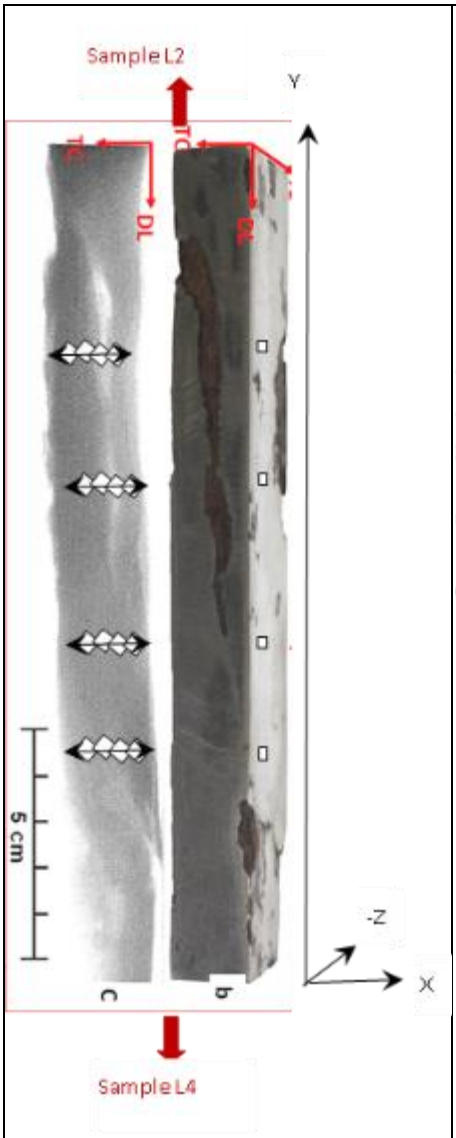

Figure 1. Iron weld. SM9 (L3 sample) Roman iron bar after surface milling. Schematic positioning of the investigated sections according to the iron sample position. The neutron beam is in the (Z,-Z) direction
(Left) X-ray view of the weld
(Right) 3D view of the welding zone.

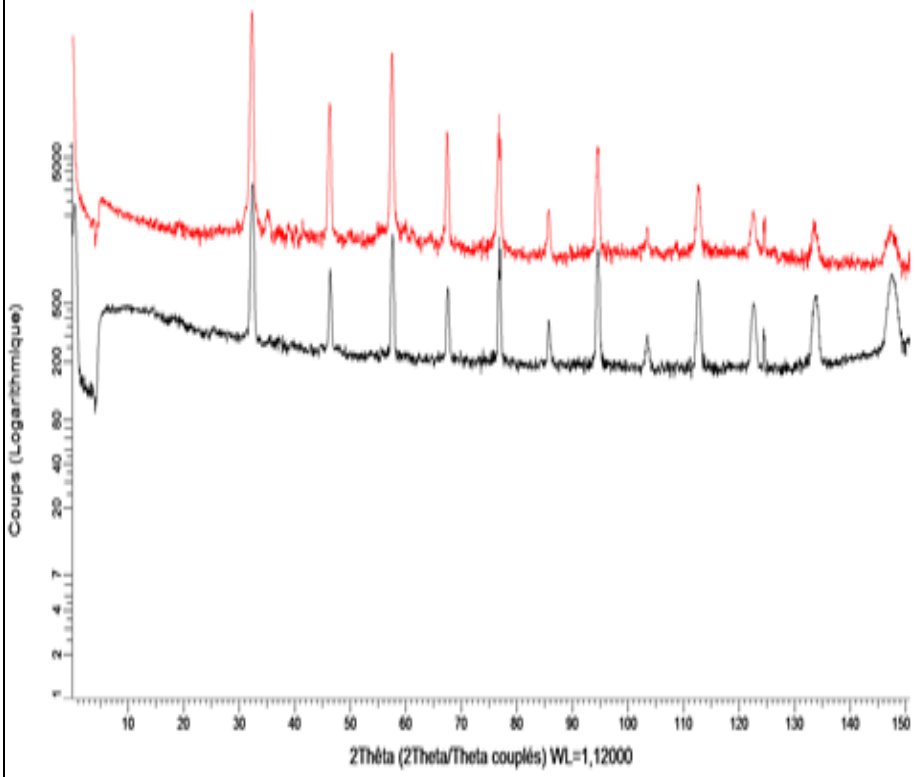

Figure 2. Two Diffractograms obtained on either side of the weld, mainly alpha iron (ferrite) matrix, and a few minor phases only on on side (red diagram). **I**ron weld: Roman iron bar SM9 L3 sample. Obtained on D20 (batch 1 conditions – cf. Table 1)

3. Improvement of experimental conditions to specific ancient metallic iron materials

In order to adapt our methodology specifically to these ancient metallic material (to improve the S/N ratio as well as to refine the diffractogram location specifically on the weld), a second batch of analysis (Table 1) was performed with a Z position closest to the incident neutrons beam (at 8mm instead of 30mm).

In a first stage, in order to check the nature of the different phases but also to identify if a lattice parameter variation of ferrite can be determined, large beam was applied (~1cm²) at different Y positions and a new set of diffractograms were collected.

In a second stage, according to these previous informative data, only two X lines were selected and scanned in order to focus on the evolution of the phases with positioning. 15 points were performed with a lower step size, a lower investigated area (small beam size) and a markedly higher counting time. Thus more defined and resolved spectra were obtained as shown Figure 3. The analyses of diffractograms are always under investigation.

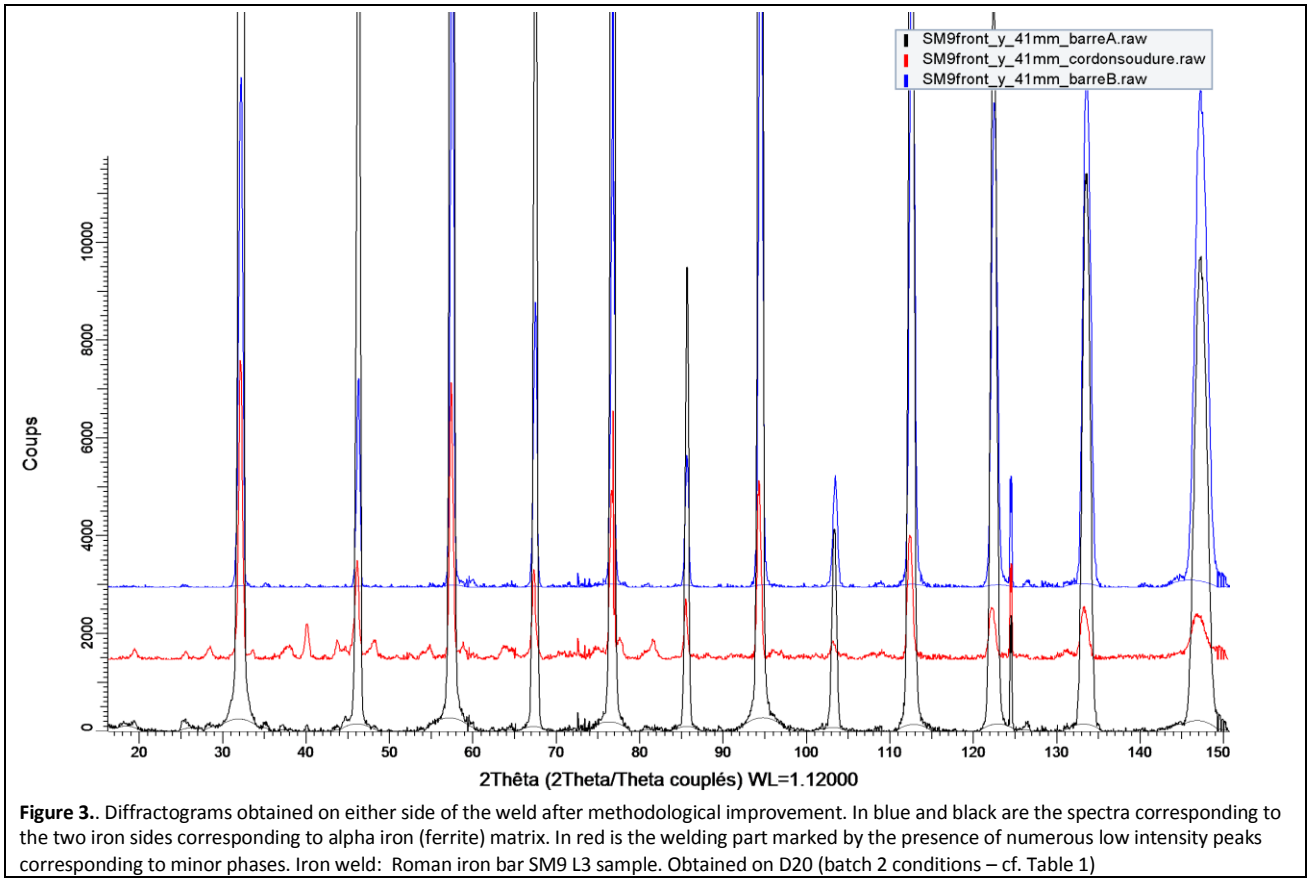

Figure 3.. Diffractograms obtained on either side of the weld after methodological improvement. In blue and black are the spectra corresponding to the two iron sides corresponding to alpha iron (ferrite) matrix. In red is the welding part marked by the presence of numerous low intensity peaks corresponding to minor phases. Iron weld: Roman iron bar SM9 L3 sample. Obtained on D20 (batch 2 conditions – cf. Table 1)

Conclusion:

Neutron diffraction was applied for the 1rst time on iron Roman bars in order to characterize the welding. In this first try, the following information were obtained:

- it is possible to identify this type of ancient and historic weld from neutrons diffraction, applied here as a non-invasive method.

- the main identified crystalline phases of the bars are only ferrite and cementite (no martensite was evidenced).

- the welding part is characterized by the presence of minor crystalline phases which can be related to oxide compounds such as silicate and iron oxides. Investigation remains under progress.

- experimental conditions have been improved and is positively available for future research.