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RESEARCH ARTICLE

CHRONOLOGICAL DATING OF THE WATER MILL ZABALE ERROTA IN PAÍS VASCO (SPAIN)

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ABSTRACT

In this work presented aims chronological dating of a water mill found in the País Vasco (Spain) by quantitative metallography of the natural aging process of the steel over time, for possible protection and restoration thereof.

INTRODUCTION

Until the mid-twentieth century, the mill under study formed a unique property with Zabale village that had been dated to the sixteenth century. At present, it is unknown if the mill is before or after the settlement. The mill has remained active until the mid-twentieth century and inhabited until 1967, when he suffered a fire that destroyed his cover. So far, it has remained so no one has taken charge of its restoration, so not on the inventory of mills in the País Vasco (Spain), even though the village Zabale yes it is. The mill has happened to call Zabale-Errota (Figure.1 & 2)

Figure.1 Image of the current view of the water mill Zabale-Errota.

Figure.2 Map of the current location of the water mill and the village Zabale.

The Zabale-Errota mill is fed by the waters of the stream Zubitxu, leading to the beach Górliz. In order to achieve this will be included in the list of mills to be protected by the Basque Government and thus obtain funding for restoration as closely as possible, your dating is necessary. Its possible classification as a cultural inventory, or cataloged, promote recovery.

To facilitate this classification was made a historical study, but found the oldest reference is from 1768, in a marriage contract. However, it is suspected that the mill

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could be older. For what is done should one archaeometric study of the building.

Was used to analysis by C-14, but was unsuccessful by industrial pollution of the País Vasco in recent centuries.

Same happened with thermoluminescence technique because sensitive materials technology: tiles, bricks, etc., were affected by the fire of 1967, or were added after the initial work done all of it with masonry.

We also attempted to dendrochronology, but the beams salvaged from the fire had a small number of rings to use the technique mentioned.

Finally, the dating was used by quantitative metallography of the natural aging process of the steel over time (Chadwick, (1972); Ree-Hill, (1973); Porter, (1981); Criado *et al,* (2000); Criado *et al*, (2004); Criado *et al*, (2006); Peña-Poza *et al*, (2010); Criado Martín *et al,* (2013)). This method is based on changes in the microstructure produced by the slow diffusion of ferrite will contour, over long periods of time, such as hundreds and thousands. These morphology changes are readily observable in perlite crystals in which a reasonable change is seen in the form of sheets cementite (Criado *et al*, (2000); Criado *et al*, (2004); Criado *et al,* (2006)). Ferrite crystals in the diffusion processes which cause a change is not so, but a stress relieving, so it becomes easier to measure its resistance loss by microhardness. All these changes are observable and measurable, especially in carbon steels hypoeutectoid hot forged and normalized, which constitute the majority of the artifacts steels, the model was published by Criado *et al.* (Criado *et al*, (2004)).

In the field study of the water mill Zabale-Errota presence was observed in an older stratigraphy, prior to its use as a flour mill, the presence of slag, which were also examined. The collection of such slags was made according to the method of Harris Matrix (Harris, (1989)) (Figure.3).

Figure.3 Macrograph some of the slag collection oldest mill construction Zabale- Errota which were examined stratum.

Experimental Technique The bars of the windows several pieces with abundant metallic core were cut (Figure.4 & 5). Various samples were mounted in two-component resin Rèsine Mècaprex KM-U by Presi and subsequently sanded and polished conventionally with Buehler abrasive paper with a grain of 120, 270, 320 and600; and a Buehler polishing cloth with alumina α (0.3μm) by Buehler. Etching was carried out with 4% Nital (Figure.6) in ultrasonic bath for 90 seconds. They were observed through a conventional optical microscope and areas that had ferrite crystals of suitable size, so that the traces of microhardness were not affected by cristale perlite were selected.

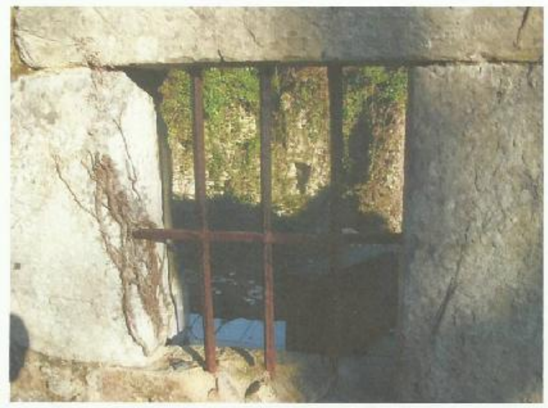


Figure.4 Image showing the state of the iron bars taken is observed to take samples of the mill Zabale-Errota.

Figure.5 Image samples collected some of the bars Zabale-Errota mill.

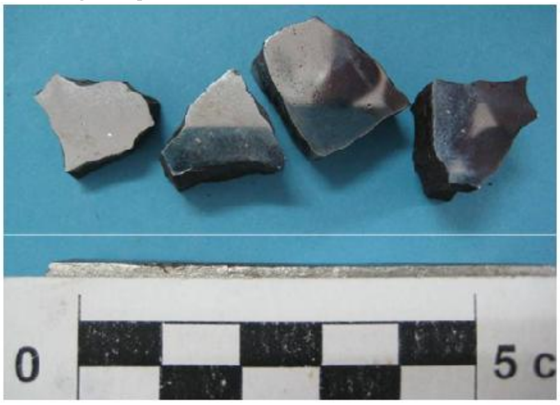


Figure.6 Macrography showing some of the specimens prepared from samples collected.

Then, we proceed to measure their microhardness Vickers with a microhardness instrument Akashi MVK-E3 model with built-in video camera under DIN EN ISO 6507-1: 2005 and ASTM E-384 (Figure.7).

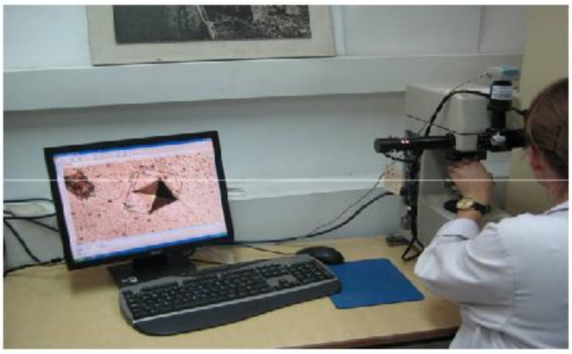


Figure.7 Image of microhardness instrument built-in video camera used in research.

The hardness measurement was conducted in the captured images on a computer screen with PEO- microdurometer program AxioVision Release-4.8.2. Ten measurements on each of the four samples were made ready. In total, 40 tracks of hardness, which were measured accurately made. After statistical study of them, an average value, which was used to compare with the linear regression line derived proposed by Criado *et al* model was obtained. (Figure.8).

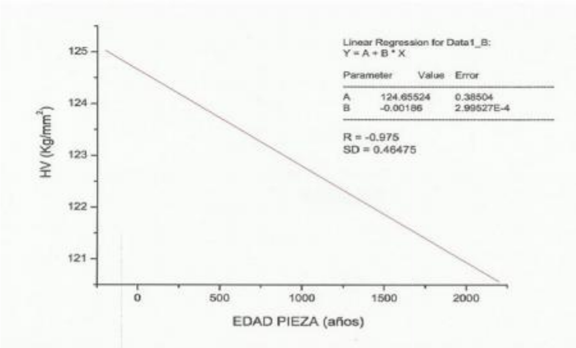


Figure.8 Linear regressi model published by Criado *et al.*

RESULTS AND DISCUSSION

Traces of hardness were you throw all of them in quite rude ferrite crystals, which was not difficult; since it was iron with a carbon content very low, below 0.1% carbon by mass (Figure.9).

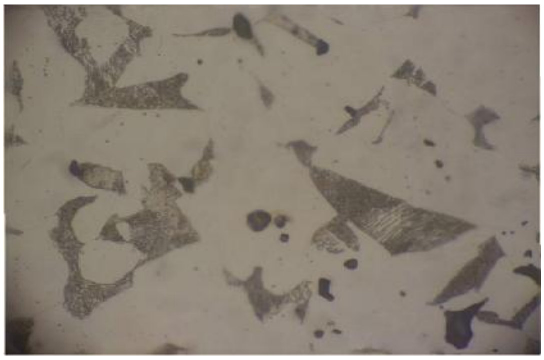


Figure.9 Micrograph showing the microstructure of the steel bars. It is a hypoeutectoid steel low carbon content.

Vickers hardness values fluctuated in a narrow range, as shown in tables I and II (Figure.10).

Table I Table of hardness measurements of the samples tested with traces diameters.

	HV	F, Kg	d1, µm	d2, µm	dm, µm
1	124.0	0.05	27.13	27.56	0.027345
2	126.0	0.05	27.34	26.9	0.027120
3	121.7	0.05	28.16	27.02	0.027590
4	124.0	0.05	27.16	27.53	0.027345
5	124.3	0.05	27.33	27.29	0.027310
6	118.6	0.05	28.15	27.77	0.027960
7	126.9	0.05	27.07	26.99	0.027030
8	118.5	0.05	28.12	27.77	0.027940
9	126.2	0.05	27.07	27.14	0.027105
10	125.7	0.05	26.88	27.43	0.027155
Middle value	123.6				

Table II Table with hardness measurements, the samples 1, 2, 3 and 4

	SAMPLE -1 HV0.5Kg//g	SAMPLE -2 HV0.5Kg//g	SAMPLE -3 HV0.5Kg//g	SAMPLE -4 HV0.5Kg//g
1	124.0	126.1	126.5	125.9
2	126.0	121.6	120.0	126.3
3	121.7	118.4	125.7	121.1
4	124.0	127.1	125.6	123.2
5	124.3	125.4	124.3	123.8
6	118.6	118.8	123.9	125.8
7	126.9	125.9	124.0	125.3
8	118.5	124.6	122.7	123.7
9	126.2	124.3	124.2	122.4
10	125.7	123.3	120.3	120.6
Middle value	**123.6**	**123.5**	**123.7**	**123.8**

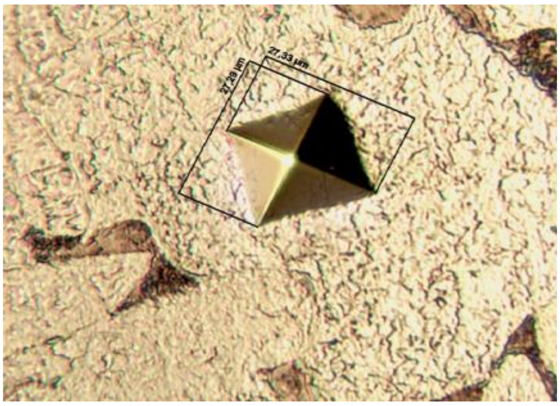
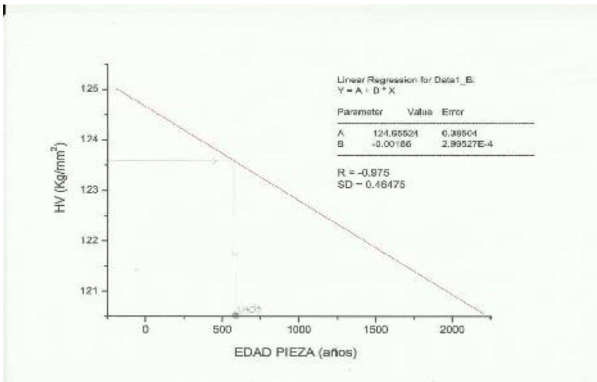


Figure.10 Micrograph showing a microhardness traces made.

The important thing to use this method of dating is the average of all measurements obteined, which was 123.65HV, under a load of 50 grams.



Graph. 1 Straight linear regresión model published by Criado *et al.*, in which you entered the value of the HV hardness and the value obtained for dating.

Using this value, we turn to the regresión line obtained from the model published by Criado *et al*. (Criado *et al*, (2004)) (Graph.1). The value obtained for dating is 540.45 years, with an errorof ± $3 \cdot 10^{-4}$.

An examination of the found slag, following prospecting method matrix Harris, valuable information was obtained (Figure.3). The presence of these slags is very large and, alter metallographic examination by SEM with EDS-EDX análisis incorporated more specific information about their structure and Chemicals composition were obtained (Figure. 11, 12 & 13).

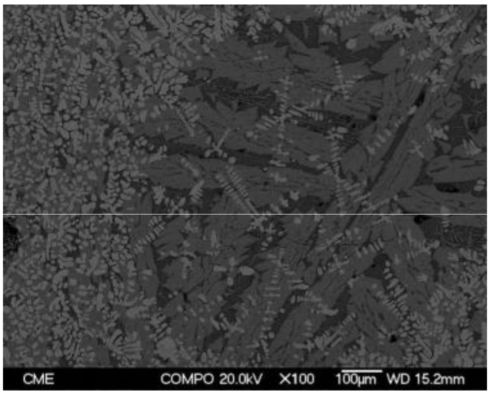


Figure.11 Calcined mineral microstructure observed by scanning electron microscopy.

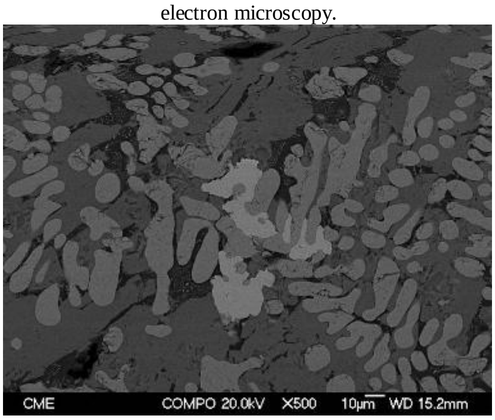
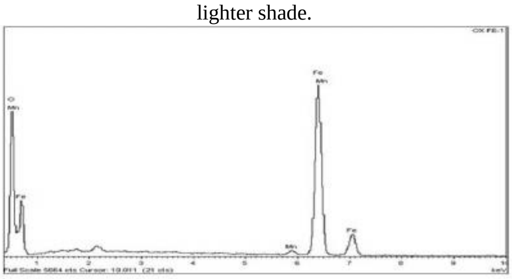


Figure.12 Calcined mineral microstructure observed by SEM with backscattered electrons, in which metallic iron is observed in the center, in lighter shade.



Element	Intensity Corrn.	Weight%	Atomic%
O K	1.6670	28.20	57.81
Mn K	0.9186	1.41	0.84
Fe K	0.9375	70.39	41.34
Totals		100.00	

Figure.13 Diffractogram EDS-EDX area in Fig.11 observed, showing a composition of iron up to 70% by mass.

It is calcined iron ore benetited in the País Vasco (Spain) since the fifteenth century. Is a calcined or siderite iron carbonate containing 70% average iron rich (Figure.13). This wealth was achieved by calcination abundant iron ore (siderite), which existed in the País Vasco (Spain) producing a mineral with the wealth to its reduction in oven. In Figure.12 shown how in some cases it came directly to metal.

In other burning materials studied, the metallic iron can be up to 30% of the resulting product.

This implies the possibility that this watermill, Zabale-Errota was used previously as installing a metal foundry (Azcárate *et al*, (1994); Díez-Saiz, (1997)). The presence of charred implies the existence of other facilities close to him, in which iron minerals (Carbonates) was calcined; forming an integral part of the old forge.

All this leads us to conclude that the installation has come down to us, but its original use was the hydraulic forging a watermill is a remarkable state of preservation; but has lost the cover of an antique which places it in the second half of the fifteenth century. Construction appears very abundant during this century and beyond (Azcárate *et al*, (1994); Díez-Saiz, (1997)).

SUMMARY AND CONCLUSIONS

The method used for dating the water mill Zabale-Errota has provided a date consistent with the existence of these facilities in the País Vasco (Spain). All results lead us to construction took place in the second half of the sixteenth century, being initially forging facility for male pylon, iron ingots obtained in the forge. This male pylon moved through the stream of Zubitxu River and later was adapted to water mill for flour.

The report was delivered to the Department of Culture of the Basque Government, which now follow relevant paperwork to name Cultural Interest. In the future it´s intended to rebuild as it has all the mechanical elements of a water mill, and used as a tourist attraction and house.

References

Azcárate, A, Palacios, V. (1994): *Arquitectura hidráulica en el valle de Cuartango (Álava)*. Molinos, Diputación Foral de Álava, Vitoria, 89-127.

Chadwick, G.A. (1972): *Metallography of phase transformations.* Butterworth and Co. Ldt., London, 187-200.

Criado Martín, A.J., García, L., Criado Portal, A.J., Dietz, C., Martínez, J.A.(2013): *Morfology of the cementite in archaeological steels that have suffered fire*, Revista de Metalurgia, 49 (4), 257-265.

Criado, A.J., Martínez, J.A., Calabrés, R., Karlsson, M. (2000): *Microstructures in historial and archaeological steel objects o resulting from aging process*, Praktische Metallographie, 37 (2000) 6, 315-325.

Criado, A.J., Martínez, J.A., Dietz, C., Bravo, E., Criado, A.Javier, Arévalo, R., Jiménez, J.M.: (2004): *A new method for datation of ancient steel simples using Vickers microhardness*, Materials Characterization, 52, 145-151.

Criado, A.J., Martínez, J.A., Dietz, C., Criado, A.Javier, Arévalo, R., Guzmán, J., Chamón, J., Bravo, E.: (2006): *Archaeologic analogues: microstructural changes by natural ageing in carbon steels,* Journal of Nuclear Materials, 349, 1-5.

Díez Saíz, A. (1997): *Molinos de río en el valle de Asua (Bizkaia)*, Eusko Ikaskuntza. Zainak. Cuadernos de antropología etnografía, 14, San Sebastián, 263-318.

Harris, E.C.(1989): *Principles of archaeological satigraphy*, Second Edition, Academic Press Limited, London.

Porter, D.A., Easterling, K.E.(1981): *Phase transformations in metals and alloys*, Edit. Van Nostrand Reinhold Company, London, 279-287.

Ree-Hill, R.E.(1973): *Physical metallurgy principles,* Edit. Van Nostrand Company, New York, 661-679.

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