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Indirect Dating of a Cistern Located in a Roman Archaeological Site (1st Century BC and 1st Century AD) from a Steel Nail

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Abstract

The discovery of a steel nail in the concrete wall of a cistern of possible high-imperial Roman origin (1st century BC and 1st century AD) has made it possible to date this construction chronologically. Until now, no method of dating concrete has been possible; however, the metallographic study of this nail gives clues for chronological dating. The existence on the nail of microstructures that reveal the existence of old perlite can be a clear fact in the chronology of the steel nail and the wall where it was found inside.

Keywords: Cistern; Archeological; Steel; Cementite; Roman

Introduction

Cerro de La Coja is an exceptional archaeological site where abundant human remains from the 1st century BC and 1st century AD have been found and excavated, all associated with copper mining in Cerro Muriano (Córdoba, Spain). On the southwestern slope, there is a well in perfect condition, which has been reused until the second half of the XX century [1]. The study of concrete samples of a visible side wall (Figure 1), provided a piece of steel nail (Figure 2), belonging to the wooden formwork, which was used in its construction. A quantitative metallographic and Vickers microhardness study has been carried out on the nail piece to assess the ageing process of the steel and to estimate its age [2-4]. This is a sample that gives indirect information on the dating of the cistern.

With quantitative metallography, the process of evolution of cementite has been evaluated by diffusion mechanisms for very long periods of time of centuries and millennia. By means of Vickers microhardnesses, applied to the ferrite crystals of steel, values have been obtained that indicate the structural relaxation of this phase; in many cases, values below 100 Vickers units have been obtained [2-6].

Likewise, the data obtained from the concrete, by means of X-Ray diffraction, make it coincide with the diffractogram of the La Coja Cave reservoir, located in the same site and dated as a Roman high-imperial from the 1st century [1]. The diffractograms made on the concretes belonging to the Córdoba Copper Company (19th and 20th centuries) mining installations, present in the same deposit, differ in the content of the types of rock present.

This study will contribute to the dating of the reservoir, which will be excavated and analyzed in detail.

Experimental Technique

In a partial excavation of a side wall of the cistern under study (Figure 3), a concrete sample was taken for an X-ray diffractogram. After the sample was cut into pieces, a metal object of oxidized steel appeared inside the mass of the concrete (Figure 2). It was probably occluded during the construction of the wall. The metallographic study of this nail can give us some clues about the age of the concrete wall.

The piece of steel was embedded in resin and prepared metallographically (Figure 2); it could be verified that it was oxidized but contained a metal core. For Scanning Electron Microscopy, a gold sputtering was deposited and chemically attacked with 4% Nital.

A Vickers FUTURE-TECH model FM-700 micro-hardness tester, with a variable load of 10 kg to 100 kg, was used to determine the Vickers hardness of the ferrite phase crystals and to assess the softening of this phase by relaxation over a long period of time [3,4].

For the examination of the concrete samples, it was prepared by grinding and compared with other concrete from constructions located in the same site of Cerro de La Coja: Roman cistern called Cerro de La Coja and constructions of the Córdoba Copper Company in the same place. These samples were subjected to a diffractogram.

Figure 1: View of the excavated side wall from which the concrete sample was extracted.

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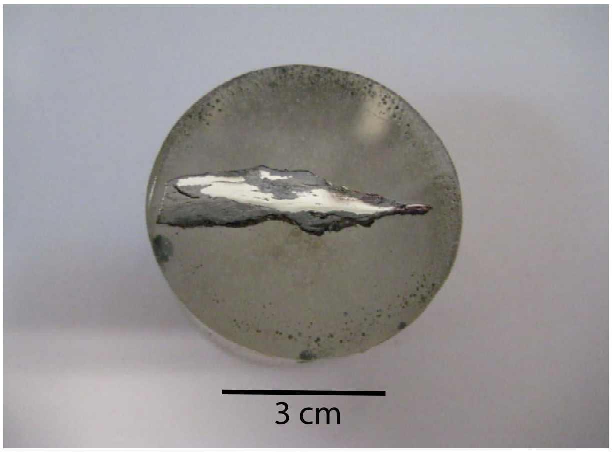

Figure 2: Concrete inlayed sample together with the piece of steel nail.

Figure 3: Situation of the cistern in Cerro de La Coja presenting a stepped wall from where the sample was extracted.

Results

The steel nail presents some metallographic keys that can provide information on the time of origin of the nail and that for us would mean data for the dating of the cistern. It is a rather heterogeneous steel nail in terms of its chemical composition in carbon. There are areas with little carbon, with crystals preferably ferritic (Figure 4).

Other areas with high carbon content are also distinguished (Figure 5). From its microstructure, it is derived that it was obtained by hot forging and air cooling. The piece of steel from which it was forged must have been a very heterogeneous piece of steel, although the piece was obtained by hot forging of different pieces of steel.

The obvious thing is that the structure of the perlite and its morphologies are not conventional. They have very straight contours and very defined cementite orientations (Figure 6). In addition, in some areas, these layers of perlite cementite appear very segmented (Figure 7). These morphologies of perlite correspond to what are called "very old pearlite" [5,7-9]. These pearlitic colonies have undergone a very long and slow aging process that has made them acquire such remarkable morphologies. For this complete changeover, a process of diffusion of carbon atoms into the ferrite matrix for long periods of time at room temperature is necessary [10-17]. There is a clear tendency to acquire elongated prismatic language forms and, in some cases, to a staggering and segmentation [18,19].

These processes are extremely slow and require millennia to conform. The present structures correspond to the approximately 2,000 years of our era.

This structural relaxation is reflected in a loss of strength of the material. This loss of strength can be quantified by Vickers microhardness [3,4].

The microhardness is related to the dating in years that normalized steel presents. Microhardness is measured in ferrite, which is the most sensitive micro constituent to the passage of time. The microhardness fingerprints were made with a FUTURE-TECH microhardness tester type FM-700, with variable load capacity from 10 g to 50 g (Figure 8 and Table 1).

The average Vickers hardness obtained is 100 HV. This value puts the steel nail more than 2,000 years old. This type of calculation can only be made with hot forged and normalized hypoeutectoid steels, which is where the relaxation and softening of the ferrite crystals can be observed and measured with the elapsed time [3,4]. The exact location of the hardness measurements on the nail is in the ferritic zone of Figure 4.

Also, the X-Ray diffraction of the cistern's concrete provided valuable parallel information compared to the diffractograms of another cistern built in the same area of the La Coja hill and perfectly dated: La Coja Cave cistern of high Roman-imperial origin with 2,000 years of age (Figures 9 and 10).

Both diffractograms could be compared and overlapped in a remarkable way (Figure 11). A concrete diffractogram was also obtained from some constructions of the same place, belonging to the English Foundry of the 19th century, showing the difference with the previous two (Figure 11).

All this indirectly points out that the two constructions, the reservoir under study and the Cueva de La Coja cistern, were built with the same type of concrete and that it could have been at the same time; since in later times, the concretes have some different characteristics.

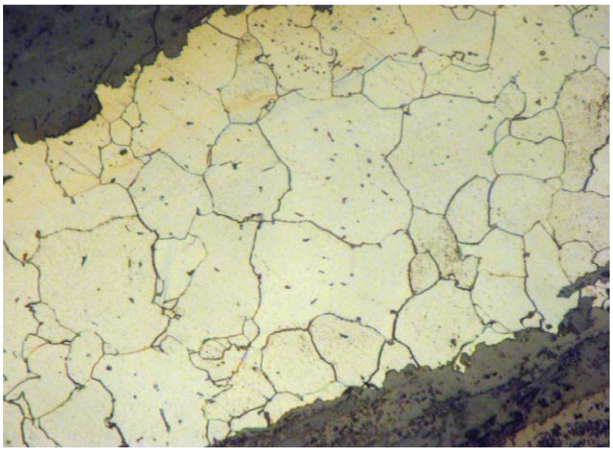

Figure 4: Micrography obtained by Conventional Optical Microscopy, in which the ferrite crystals are observed in an area of the steel nail studied.

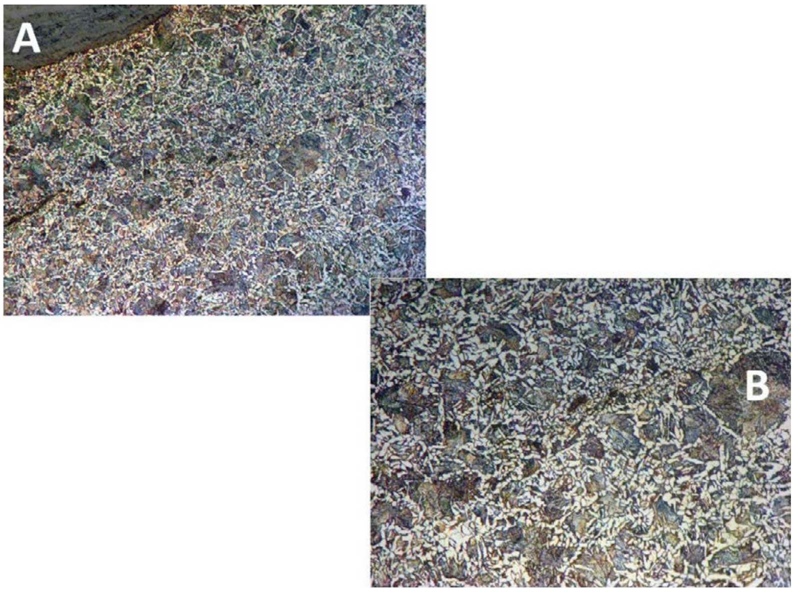

Figure 5: A) Micrography obtained by Conventional Optical Microscopy, from the area of the steel nail with a high carbon content. B) Detail at higher magnifications.

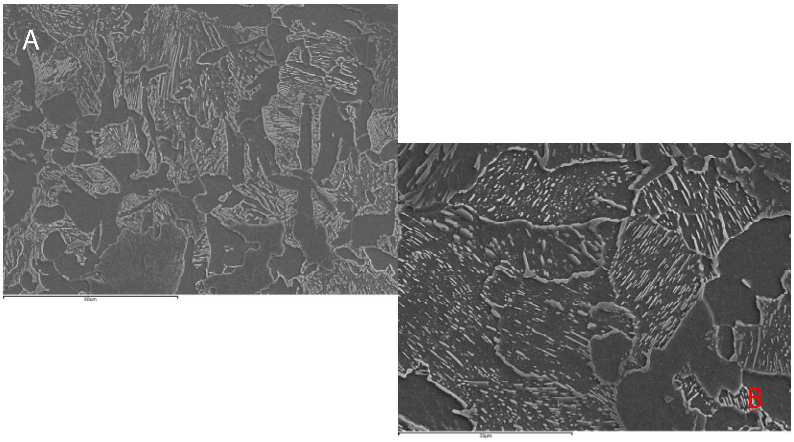

Figure 6: A) Micrograph obtained by M.E.B., where it is observed the perlite colony with sheets of prismatic linguistic cementite and oriented in all of it. **B)** Detail at higher magnifications.

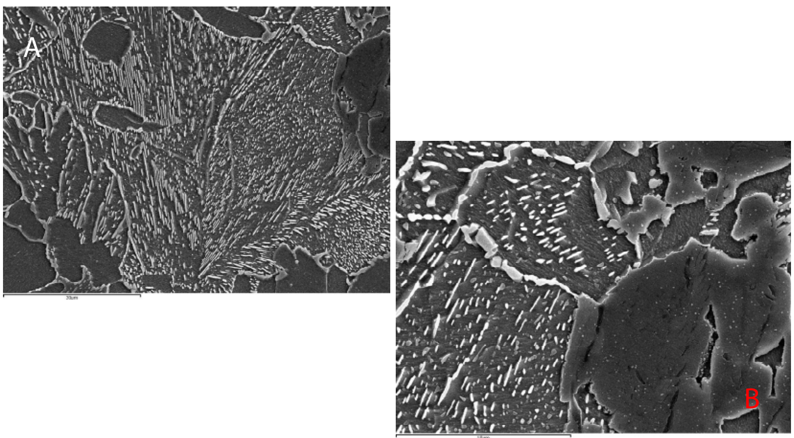

Figure 7: A) Micrograph obtained by M.E.B., where the colony of perlite with very segmented cementite is observed. **B)** Detail at higher magnifications.

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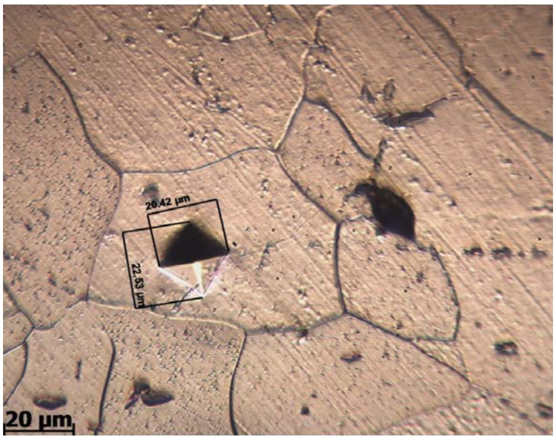
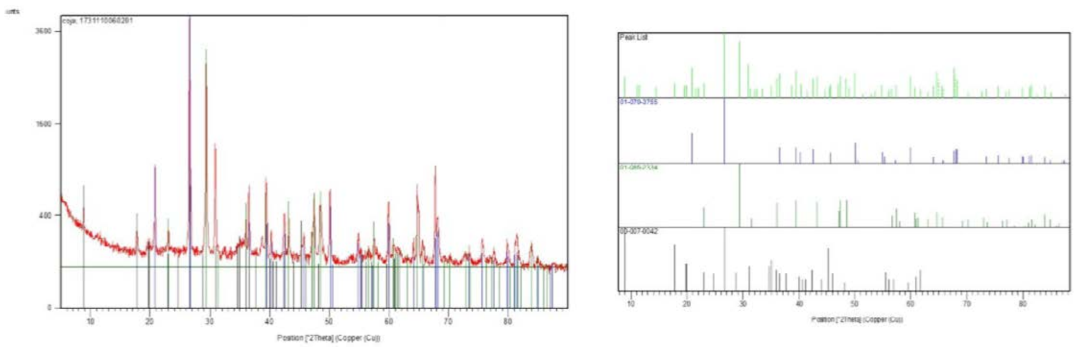


Figure 8: Micrography obtained by Conventional Optical Microscopy, from one of the fingerprints obtained with the Vickers micrometer, with a load of 25 grams.

HV (Kg/mm²)	Load (g)
90,6	25
92,5	25
108,3	25
100,5	25
103,8	25
99,6	25
100,2	25
108,4	25
99,4	25
97,6	25

Table 1: HV measurements obtained with the Vickers micrometer, with a load of 25 grams.



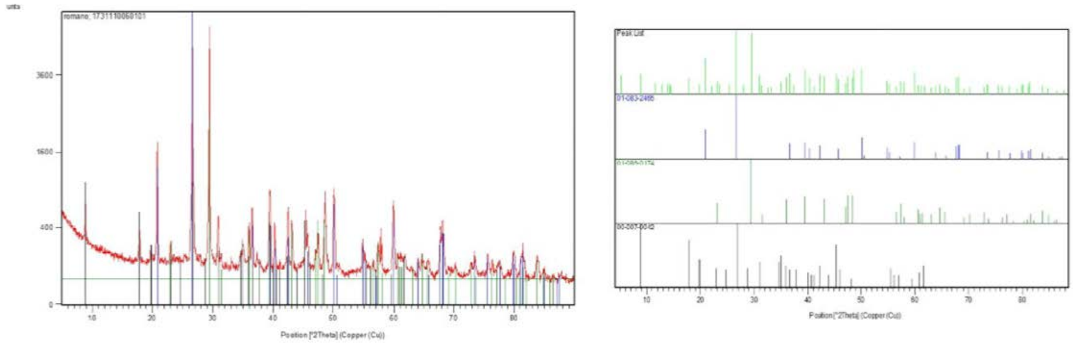
Ref. Code	Compound Name	Scale Factor	Chemical Formula
01-070-3755	Quartz	1,025	Si O2
01-086-2334	Calcite	0,756	Ca (C O3)
00-007-0042	Muscovite-3\ITT\RG	0,153	(K , Na) (Al , Mg , Fe)2 (Si3.1 Al0.9) O10 (O H)2

Figure 9: Diffractogram of the Cueva de La Coja cistern perfectly dated to 2,000 years ago; high-imperial Roman.

Conclusions

The aim is to provide data to find out the possible chronology of a well in perfect condition, located in the archaeological site called Cerro de La Coja located in Cerro Muriano (Córdoba, Spain). In order to justify an archaeological intervention in this cistern, it is necessary to provide prior information justifying this intervention. The area has an abundance of Roman constructions from the Republican and High Imperial periods, and we think that the construction of the cistern is in that period: between the 1st century BC and the first century A.D.

The analysis of the concrete led us to find in the interior of the mass, steel nail, probably belonging to the wooden formwork of its construction. The metallographic study of its steel and the



Ref. Code	Compound Name	Scale Factor	Chemical Formula
01-083-2465	Silicon dioxide $-alpha	1,031	Si O2
01-086-2334	Calcite	0,401	Ca (C O3)
00-007-0042	Muscovite-3\ITT\RG	0,166	(K , Na) (Al , Mg , Fe)2 (Si3.1 Al0.9) O10 (O H)2

Figure 10: Diffractogram of the cistern under study.

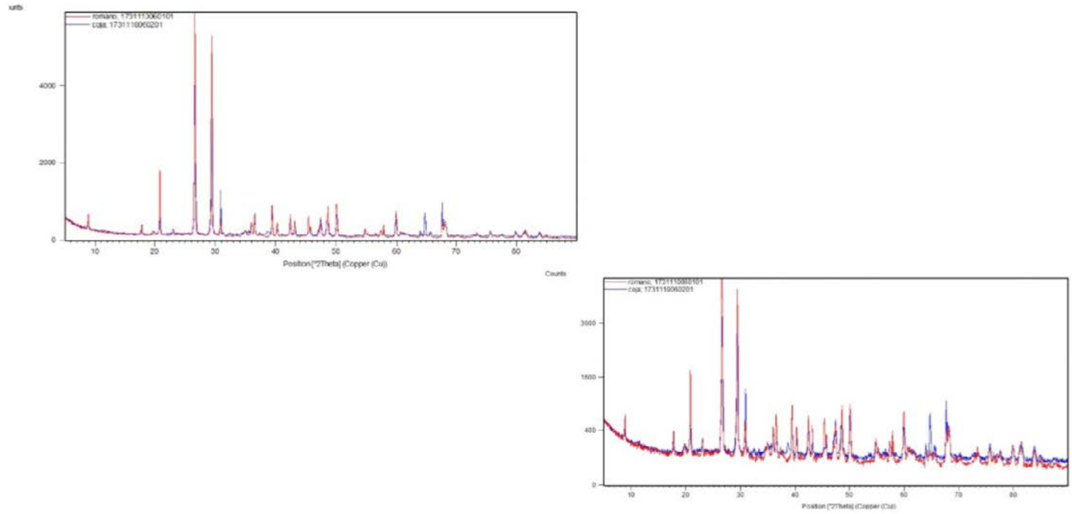


Figure 11: Upper left diffractogram: overlapping diffractograms of the cistern under study (net) and the Cueva de La Coja cistern (blue). It's a perfect match. **Right lower diffractogram:** superimposed diffractograms of the concrete of the cistern under study (network) and of the concrete of the local construction of the 19th century English Foundry (blue). Not a perfect match.

characterization of its mechanical properties, we thought they could provide some data of its age.

The presence of a very aged microstructure of the perlite, with crystals of prismatic linguistic cementite, in some cases very segmented by staggering, leads us to conclude that the time spent since its manufacture by hot forging and standardized, must have been very long. Compared to other archaeological artifact steels studied, they are very old pearlite, with a chronology of around 2,000 years.

Microhardness tests corroborate this antiquity, since the values reached by the ferrite crystals present in the sample are around 100 HV. The average Vickers hardness values, in hot forged and standardized steels, which are currently manufactured, are about 125 HV. This demonstrates the relaxation of the ferrite over such a long period of time.

Another test that may contribute to this dating is the chemical composition of the concrete, obtained by X-ray diffraction. The diffractogram obtained from the concrete of the cistern under study coincides with the diffractograms carried out on other high-imperial Roman constructions in the area of the archaeological site. However, it differs from the concrete used for the constructions, carried out in recent times, such as that of the installations of the 19th century English Foundry, carried out in the same archaeological site.

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