NEUTRON DIFFRACTION IN ARCHAEOMETRY: THE ITALIAN NEUTRON EXPERIMENTAL STATION INES@ISIS

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The Italian Neutron Experimental Station INES, located at the pulsed neutron source ISIS (U.K.), is equipped with a general-purpose neutron diffractometer that was built with a special care aiming to focus its use on archaeometric measurements. In fact, the large sample volume allows to accommodate non standard samples in the neutron beam, the diffraction banks cover an angle of almost 180°, allowing to detect the presence of texture, and the high instrument resolution enables a detailed analysis of the peak shape, to obtain information on the casting process and/or the working techniques applied during the production phase. Thanks to the high penetration power of thermal neutrons, archaeometric measurements performed through neutron diffraction allow to determine bulk properties of the sample. Here, we describe the instrument structure and present some preliminary test measurements, on standard samples, to show the high level quality of the machine. In particular, we will show some results of interest in archaeometallurgy like, for example, multiphase analysis of bronzes and quantitative detection of impurities.

INTRODUCTION

Diffraction techniques, using electrons, X-rays, or neutrons are considered as valid complements to optical or electron microscopy techniques, allowing for the assessment of phase composition of metal samples as well as the structural properties of its constituents. However, it should be stressed that electrons, due to their high charge/mass ratio, are characterized by a poor penetration power below the open surface of the sample. The situation improves, using X-rays, though their penetration power depends on their energy, with typical values going from fractions of a mm, for typical laboratory instruments, to several mm using the hard X-rays produced by a synchrotron radiation source. In this respect, thermal neutrons outperform other probes, with a penetration range of the order of several

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cm even in dense materials [1]. Thus, neutron diffraction is a powerful tool for a deep insight in material-structure and has been widely used to determine bulk properties of dense samples. This is a truly non-invasive, non-destructive technique that leaves the samples unperturbed, apart from a weak radioactive activation that expires, typically, in one-week time [2,3]. Another important difference, between neutrons and X-rays, resides in the kind of interaction with matter. Photons interact with the electronic shells and, therefore, their cross section is related to the atomic number of atoms. As a consequence, it is difficult for X-rays to distinguish between neighbouring atoms in the periodic table of the elements. On the contrary, neutrons interact directly with the nuclei, with absorption and scattering cross sections that depend almost at random on the atomic number and/or on the mass number. Thus, the neutron scattering contrast factor between neighbouring elements may be considerable and it is possible to distinguish between elements with similar atomic numbers. As a matter of fact, isotopic substitution techniques are currently used in neutron scattering to get primary information on microscopic structural analysis.
The first time of flight (TOF) neutron diffraction experiment, on samples of archaeological interest, was carried out in 2001, by Kockelmann et al. [4], studying the phase composition of archaeological potteries originating from medieval Rhenish sites.

The same technique was then extended, the following year, to the study of archaeological bronzes, carried out by Siano et al. [2], with a quantitative investigation of the multiphase composition of Etruscan bronzes. In the last five years, several scientific papers have appeared extending the application of TOF neutron diffraction technique to archaeological samples, like ceramic manufacts [5-8], metal tools [2,9-14], coins [5,15], and even marbles [16,17].

There are basically six categories of information that can be obtained using neutron diffraction: three are derived from a basic analysis of diffraction patterns and three are obtained by a careful investigation of the profile shapes and relative intensities analysis.

1) The material composition and crystal structure can be derived by determining the symmetry characteristics of the sample given by the distribution of Bragg peaks as a function of d-spacing.

2) In metal alloys, it may be possible to determine the relative concentration of components. When the lattice is mainly composed by a single atomic species and the second component is contained as an interstitial or substitution impurity (for example, tin in α-copper or carbon in pure iron), the value of the lattice parameter is a monotonic function of the guest species concentration. Therefore, it becomes an easy task to determine the guest percentage through the comparison with a calibration curve derived from standard samples of known concentration.

3) Rietveld refinement of the diffraction pattern allows determining the relative weight of the different phases present in the samples. This is a quantitative analysis that can be used, for example, to determine either the degradation level of samples through mineralization, or the temperature of the fabrication process.

4) Sample homogeneity analysis can be inferred by checking the shape of the Bragg peaks. Sharp peaks are characteristic of homogeneous samples that have been processed in a slow thermal annealing. On the contrary, hardly hammered or fast-cooled samples give rise to different peak shapes, peak-broadening, and/or side-peaks, due to the shift of slip planes and the formation of dendrites.

5) The possibility of rotating the sample, coupled with the availability of a high number of detectors, allows to determine the relative intensity of the Bragg peaks as a function of the rotation and scattering angle, thus giving information on the crystallites preferred orientations (texture). The shape of textures, projected on a polar plane, gives information on the working techniques and the rolling direction of the sample.

6) By analysing the Bragg peak shape on detectors placed on opposite sites (i.e. scattering angles $2\theta = \pm 90°$), with the sample oriented at $\varphi = 45°$ with respect to the incident neutron beam, it is possible to determine residual stresses. The difference between the diffraction patterns can be used to determine structural differences along orthogonal axis, thus identifying axial or planar strains.

The subsequent part of the paper is organised as it follows. In the first Section, we present the INES layout and its characteristics, as well as a short report on first year activity. In Section 2 we will discuss some standard measurements performed to determine the quality level of the instrument. The third Section will be devoted to discuss some measurements performed on samples of archaeological interest and to show some preliminary results. In the last Section we will draw some conclusions and discuss possible developments.

THE INES STRUCTURE AND ITS USE

The Italian Neutron Experimental Station, INES, is located at the world most powerful pulsed neutron source (ISIS, UK) and is operative since beginning of 2006. During this year, 25 experiments were selected by a peer review panel, and were actually executed for a total beam time of 123 days. In its present configuration INES has been widely used by the Italian and international scientific community to obtain high-resolution neutron diffraction patterns of cultural heritage artefacts. The fraction of beam-time allocated for archaeometry-related measurements, turned out as high as 50% of the total.

The neutron TOF diffractometer is equipped with 144 3He neutron detectors, grouped into 9 banks, deployed on a horizontal plane, at 1 meter distance from the sample position. The overall range of scattering angles extends from 9° to 171°. The resolving power is very good, especially for the back scattering detector banks. The sample container has been designed so that any ISIS standard sample environment equipment (i.e. furnaces or cryostats) can be adapted. This is made of a large vacuum tank (800 mm diameter by 900 mm height) which allows for the experimental investigation of almost any kind of object, including bulky archaeological artifacts. In addition, the sample tank is equipped with four optical windows, where web cameras can be adapted for a visual inspection of the sample during the experiment. A diode laser device is available to visualise the centre of the incident neutron beam for objects of irregular shape. The neutron beam section, at sample position, is a 38mm x 38mm square, with a very uniform intensity distribution. In Fig. 1, we show a 3D rendering image of the instrument layout as well as a picture of the beam profile.

The 13 archaeometry-related experiments, performed during 2006, cover a large range of different materials, going from ancient paper, to pottery, organic samples (teeth and bones), and metals. Samples of interest for archaemetallurgy range from silver and bronze coins, to bronze and iron weapons, bronze tools, and to a series of bronze standard samples with tin weight ranging from 0 to 20% and different annealing grades. Experiments range from non-destructive bulk phase analysis of bronzes, to residual stress analysis of metal objects, to texture analysis of coins and metal tools. The possibility to separately analyse each single detector makes texture analysis quick and easy. Some preliminary results on samples, where the authors were directly involved, will be shown in the third part of this work. These are related to phase and composition analysis of calibration bronze standard samples and a phase analysis on a bronze manufact.

STANDARD MEASUREMENTS
AND INES CHARACTERIZATION

The INES diffractometer is located at the end of a neutron beam-line, looking at the ambient water moderator, at a distance of 22.8 meters from the pulsed neutron source. Neutrons are produced in a white beam, with a frequency of 50Hz, allowing for TOF measurements triggered by the spallation event of fast protons on the nuclear target. Each 3He neutron detector provides a full diffraction pattern whose d-spacing span and resolution depends (apart from the energy-time shape of the primary beam) on its position with respect to the sample and the scattering angle. For the sake of simplicity, the recorded data are collected in 9 groups, each related to
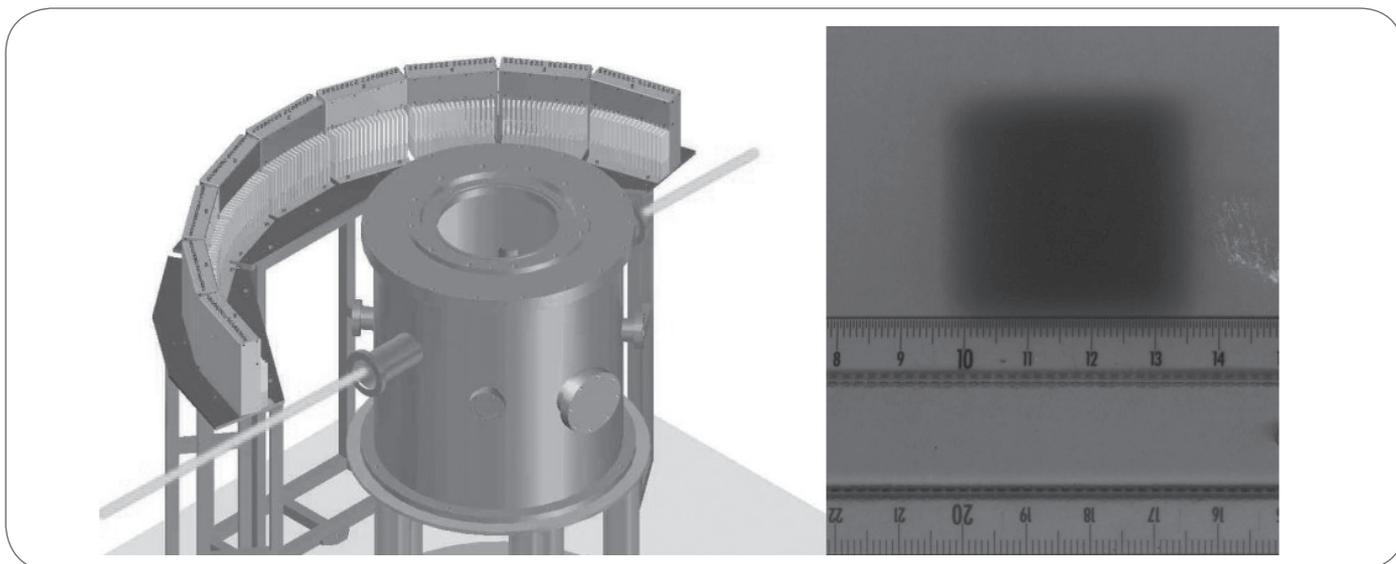
Left panel: INES layout showing the sample chamber (the primary neutron beam, green in the picture, goes from left to right) and the nine detector banks, spanning an angular range between 9° and 171°. Right panel: photographic picture of the neutron beam, taken at sample position, showing its uniform and regular shape.
Figura di sinistra: schema di INES che mostra la camera campione (il fascio primario dei neutroni, in verde nel disegno, va da sinistra a destra) e i nove banchi dei rivelatori che coprono un intervallo angolare fra 9° e 171°. Figura di destra: immagine fotografica del fascio di neutroni, presa nella posizione del campione, che mostra la sua distribuzione uniforme e la sua forma regolare.

the data of a 16-detectors bank. The d-spacing interval and the resolution of a representative sample of detectors are reported in Tab. 1. We observe how the resolving power of the instruments is very high in the d-spacing range of interest for archaeometallurgy (0.1-3.0 Å). The data reported in Tab. 1 were obtained by measuring the diffraction patterns of a standard Si 640c powder sample that is shown in Fig. 2.

Basing on several test experiments carried out on the instrument, we were able to determine a reasonable minimum time-interval allowing for a good quality measurement. This value is strongly dependent on the nature, thickness, and shape of the samples. However, for square shaped bronze samples with a thickness of 5 mm, the time needed to perform a good quality experiment is about 3-4 hours.

Detector Label	2θ (°)	Q_{min} (Å^{-1})	Q_{max} (Å^{-1})	d_{max} (Å)	d_{min} (Å)	$\Delta d/d$
A-1	170.6	3.85	62.62	1.63	0.10	0.0012
B-1	152.6	3.76	61.04	1.67	0.10	0.0012
C-1	134.6	3.57	57.96	1.76	0.11	0.0013
D-1	116.6	3.29	53.45	1.91	0.12	0.0015
E-1	98.6	2.93	47.62	2.14	0.13	0.0017
F-1	80.6	2.50	40.62	2.51	0.15	0.0020
G-1	62.6	2.01	32.63	3.13	0.19	0.0025
H-1	44.6	1.47	23.82	4.29	0.26	0.0035
I-1	26.6	0.89	14.44	7.07	0.44	0.0056
I-16	11.6	0.39	6.33	16.13	0.99	0.0130

Scattering angles, ranges of momentum transfer and d-spacing, and resolution of a number of sample detectors. The alphabetic characters (A, B, C,…) label the bank, while the numbers (1, 2, 3, …) label the detectors. For the last bank (I) we report the relevant values for the first (1) and the last (16) detector.
Angoli di scattering, intervallo del momento trasferito, intervallo spaziale di diffrazione e risoluzione di alcuni rivelatori. Il carattere alfabetico (A, B, C, …) indica il banco mentre i numeri (1, 2, 3, …) indicano i rivelatori. Per l'ultimo banco (I) riportiamo i valori significativi per il primo (1) e l'ultimo (16) rivelatore.
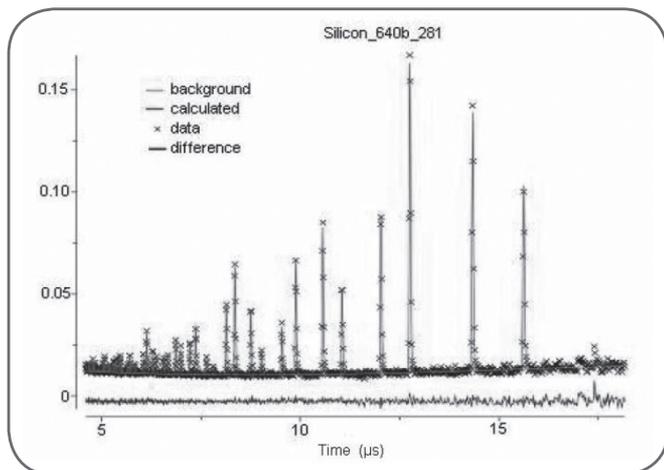
Diffraction pattern of a standard Si 640c powder sample. It is worthwhile to observe the very low background level and the high quality of the peak shapes.

Figura di diffrazione di un campione in polvere di silicio standard Si 640c. E' interessante osservare il livello del fondo molto basso e l'elevata qualità dei picchi di diffrazione.

Lattice parameter distribution, as a function of tin concentration, in copper α-phase. Data obtained by XRD are from ref. 18.

Distribuzione del parametro reticolare in funzione della concentrazione in peso di stagno nella fase α del rame. I risultati osservati tramite diffrazione di raggi x vengono dal riferimento 18.

ARCHAEOLOGICAL SAMPLES: PRELIMINARY RESULTS

This section is devoted to the presentation of preliminary results obtained in the analysis of metal samples performed with neutron scattering. To this aim, it is important to recall other common analytical techniques that are generally applied in these cases.

Techniques based on the optical and electronic microscopy (as for example metallographic analysis, SEM-EDX and XRF) are quantitative and generally very accurate. However, they only give information on a very small area that is rarely fully representative of the average characteristics of the sample. In addition, the specimen to be analysed frequently needs to be sampled or, at least, preliminarily worked. These techniques, when used to derive quantitative information, are based on an atomic analysis method so that it is rather simple to obtain the relative concentration elemental composition. On the other hand, neutron diffraction (and XRD, for some aspects) gives quantitative information of phase analysis over the scattering volume that, in our case, varies from 38x38xd mm3 down to 4x4xd mm3, where d is the sample thickness in mm. The resulting multiphase analysis can be performed on complex materials allowing to quantify the presence of pure metal elements, alloys, and corrosion products. We point out that when using Neutron Diffraction (ND) techniques the specimen doesn't need to be sampled, or preliminarily worked, and this technique can be considered as totally not destructive and not invasive. In fact, the relative concentration of the components in a binary alloy can be easily obtained from the measurement of the lattice parameters, once a calibration curve is provided. In addition, minor components in the sample are revealed from a careful analysis of their characteristic Bragg peaks revealed in the diffraction figure. The detection limit changes as a function of the scattering power of the different phases. However, we have been able to detect minor components to a lower value of about 0.1% with measurements extending for 5-6 hours
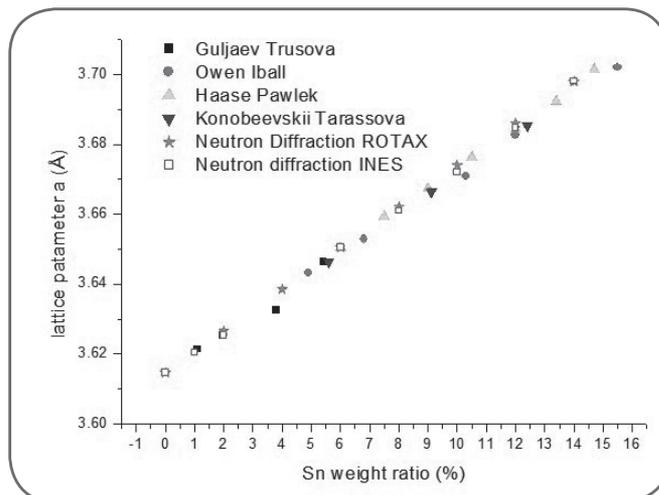
with sample volumes of about 20x20x20 mm³.

To give an example of the method, we report here the binary bronze calibration curve for the lattice parameter of the copper α-phase containing tin impurities. To this aim, we produced several (certified) reference samples, with different tin concentration, ranging between 0 and 14 wt %, in copper. From the measured diffraction patterns, it was possible to derive the lattice parameters of the fcc cell, that turned out to change as function of tin content. Starting from the pure copper value, we observe an increase of the lattice parameter, which is linear with tin concentration in the measured range. This result is not new and had been already obtained using the neutron diffractometer ROTAX [3]. However, the present results, shown in Fig. 3, were obtained with a much higher quality of the measured data (cf. Fig. 5 of Ref. 3).

Another interesting example is the possibility of analysing the bulk composition of the dendritic rich materials of a non-annealed sample. In fact, a bronze standard specimen, containing 12% tin weight concentration, was divided in two half, one of which was left as cast, while the second one was annealed for two hours in an oven at 700ºC. The former specimen turned out to be characterized by the presence of an extensive dendritic pattern (see the Scanning Electron Microscopy picture in Fig. 4). The different concentrations of the various components of the phase were evidenced by ESEM-EDX surface analysis showing two main different phase concentrations, namely a tin-rich phase (15 wt % Sn, light-gray in Fig. 4) and a copper-rich phase (4.5 wt % Sn, dark-gray in Fig. 4). Beyond these two main components, some black areas are visible on the surface (due to local fractures) and a small area very rich in tin (~25 wt % Sn, almost white-coloured in centre of Fig. 4). On the contrary, the same analysis, carried out on the annealed half, gives a homogeneous pattern with 12% Sn weight concentration, i.e. equal to the nominal title. It is self-evident that a non-destructive evaluation of the bulk dendritic composition of the as-cast specimen is almost impossible.
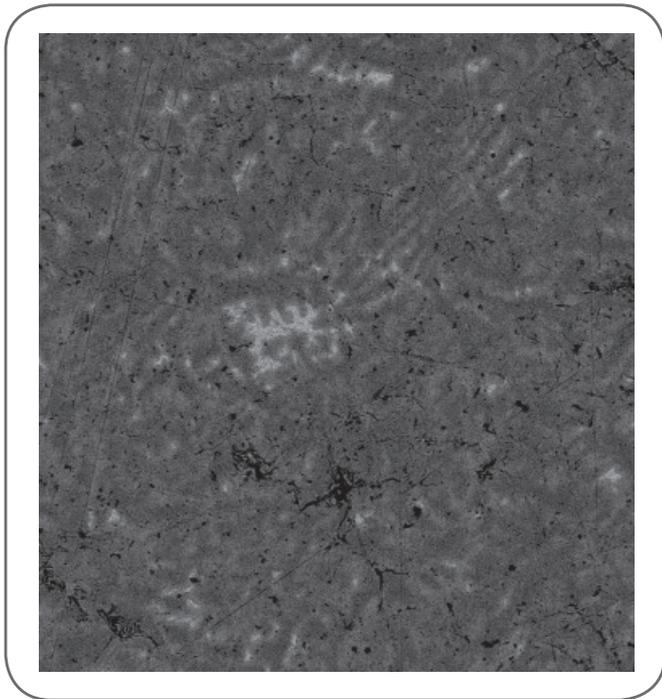
Electron Microscopy image of a cast bronze sample containing 12 wt % Sn and showing a dendritic structure. The light-coloured islands are characteristic of a higher tin concentration (15 wt % Sn) while the dark ones are more rich in copper (4.5 wt % Sn).
Immagine al microscopio elettronico (SEM) di un campione di bronzo as cast contenente il 12% in peso di stagno che mostra una forte struttura dendritica. Le isole di colore chiaro sono caratteristiche di una maggiore concentrazione di stagno (15% Sn in peso) mentre quelle scure sono più ricche in rame (4.5% Sn in peso).

A neutron diffraction analysis of the same as-cast sample evidenced a double pattern of Bragg peaks of the same crystal symmetry [F m 3 m] but linked to a distribution of different tin concentrations. This originates from an internal sub-distribution of tin concentration around two main values. Through Rietveld analysis of the diffraction patterns, it was possible to gain information on the different lattice parameters and these data were compared with the experimental results coming from the annealed half. As an example ,we report in Fig. 5 the
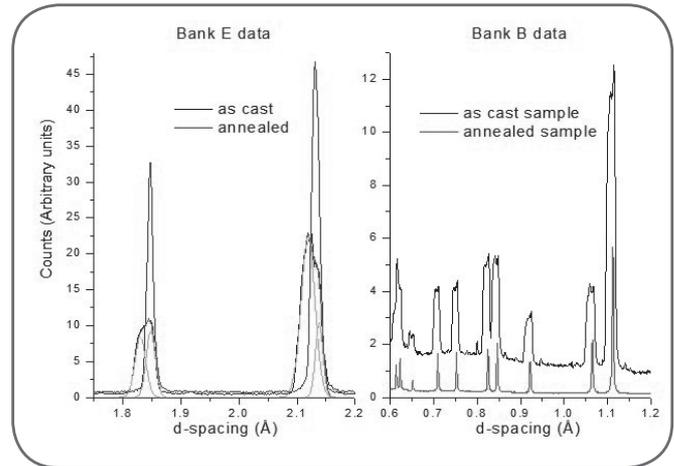
Diffraction patterns of the as-cast bronze alloy compared with the annealed one. In the diffraction pattern from the bank-E (left panel) two compositions are evidenced by the gaussian double peaks fit. The same happens in the bank-B pattern (right panel), the fit is not reported to evidence the good resolution level. *Figure di diffrazione di un campione di bronzo as cast paragonato con uno omogeneizzato. Nella figura di diffrazione del banco E (lato sinistro) sono presenti due distinte figure di diffrazione che rappresentano due diverse composizioni evidenziate dai picchi doppi. Lo steso avviene per la figura di diffrazione del banco B (lato destro), il fit dei dati sperimentali non è riportato per mettere in evidenza il livello di risoluzione delle misure.*

measured diffraction patterns for two representative detector banks. The high-resolution signal from the annealed sample of the back-scattering detector bank (labelled as B in Fig. 5) gives an idea of the instrument resolving power. By comparing this result (red pattern in the right panel) with that produced by the other specimen (black pattern in the right panel) it is possible to infer the presence of two broad distributions of concentration in the as-cast sample. The same happens looking at the pattern from the forward-scattering bank, where each Bragg peak is fitted using two Gaussian distributions whose width is larger than that of the annealed sample.

To sum up, only two main components are found to be present in the as-cast specimen, each characterized by its own average tin concentration and distribution. The measured values are reported in Tab. 2. It is worthwhile noting that the two concen-

200 peak position (Å)	Lattice parameter a (Å)	Sn wt %	Gaussian half width	Annealed sample Sn wt %
1.830±0.009	3.660±0.018	7.0	0.2	12.0
1.848±0.008	3.695±0.015	13.1	0.3	

Determination of the Sn content (weight %) of the two components present in the as-cast bronze alloy by using the different values of the lattice parameters. The different Gaussian half-width values are due to different distributions around the average value.
Determinazione del contenuto percentuale in peso di stagno dei due componenti presenti nella lega di bronzo as cast utilizzando i diversi valori del passo reticolare. Le diverse larghezze a metà altezza dei picchi Gaussiani sono dovute a una diversa distribuzione di concentrazione rispetto ai valori medi.
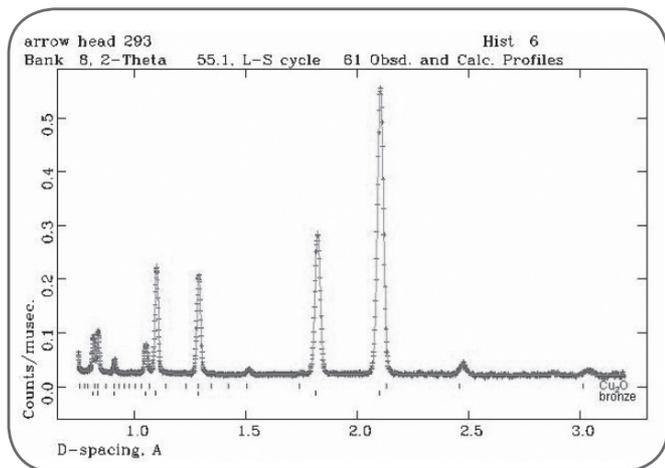
▲ Fig. 6

Diffraction pattern of a bronze archaeologic finding (arrow head). From the lattice parameter (a = 3.641 Å) we could deduce the actual composition of the alloy (4.8 wt % Sn). The presence of a not negligible amount (1.6 wt %) of cuprite impurities is also obtained from Rietveld refinement.

Figura di diffrazione di un bronzo archeologico (punta di freccia). Dal parametro reticolare (a = 3.641 Å) si può ricavare la composizione della lega (4.8% Sn in peso). E' stata determinata anche la presenza di una quantità non trascurabile (1.6% in peso) di cuprite.

tration distributions are different, with the tin-rich component relatively narrower than the other one.

Another interesting application of Rietveld refinement of neutron diffraction patterns resides in the possibility of obtaining multiphase analysis of samples and showing the relative abundance of the various phases, including bulk impurities. As an example, we show here a multiphase analysis performed on an historic bronze (arrow head from central Italy) evidencing a bulk bronze composition with 4.8 wt% tin and the presence of a considerable amount of cuprite impurities (both on the external surface and in the internal cavities) due to environmental aging (the actual positioning of cuprite cannot be distinguished by ND and needs to be evidenced by microscopic techniques).

In the diffraction pattern reported in Fig.6, we evidence the presence of two different patterns ascribed to two distinct phases: copper-bronze fcc phase and cuprite phase. The quantitative analysis of the relative intensities of the two diffraction patterns gives their relative weight percentage over the sample volume, 30x10x8 mm^3, that was totally irradiated by the neutron beam. The relative concentration of tin has been determined through the knowledge of the calibration curve of the copper-tin alpha phase alloy. It is important to note that, by this method, the presence of other elements (like zinc, or arsenic) inside the alloy cannot be distinguished and other independent techniques (e.g. qualitative analysis) needs to be applied in order to complete the information about the sample composition.

CONCLUSIONS

In this short report, we have presented the most relevant features of the Italian Neutron Experimental Station, INES, that is operated by the Italian Consiglio Nazionale delle Ricerche and is located at the world most powerful pulsed neutron source ISIS (UK).

INES is equipped with a neutron diffraction instrument which represents a valid and powerful tool to look inside dense matter and to obtain a wide range of information on its microscopic properties. In particular, for metals of archaeological interest, it is possible to gain information on the phase composition of samples that is averaged on the scattering volume, in an absolutely non-invasive way. The data, once analysed and interpreted, can give useful information on manufacturing processes, structural properties, and degree of conservation. It is important to stress, once more, that the kind of information obtained by neutron diffraction is averaged on the bulk of the sample and is not limited to the surface and the region close to it. The data analysis is performed on diffraction patterns presented in time of flight (microseconds scale) or d-spacing (in nm or Angstrom) using Rietveld refinement for multiphase analysis technique exploiting public domain software (like for example GSAS) especially developed to this aim. The combined use of neutron diffraction with other non-destructive techniques such as radiography, x-ray fluorescence, x-ray diffraction and optic spectroscopy can cover almost all the range of interest for non-destructive archaeometric analysis, thus avoiding any sampling and the consequent degradation of precious items.

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ABSTRACT

LA DIFFRAZIONE DI NEUTRONI IN ARCHEOMETRIA: LA STAZIONE SPERIMENTALE ITALIANA PER NEUTRONI INES@ISIS

Parole chiave: caratterizz. materiali, diffrattometria, prove non distruttive

La stazione sperimentale italiana per neutroni INES, situata presso la sorgente pulsata di neutroni ISIS (GB), è equipaggiata con un diffrattometro a neutroni altamente versatile che è stato realizzato con particolare attenzione per quanto riguarda le misure di carattere archeometrico. Infatti, sia l'elevato volume della zona campione che consente di posizionare campioni non standard nel fascio di neutroni, sia la geometria dei banchi di diffrazione che coprono un angolo di quasi 180° sul piano orizzontale che permettono di individuare la presenza di tessitura nei campioni, sia l'elevata risoluzione strumentale che consente l'analisi dettagliata del profilo dei picchi di diffrazione, consentono di ottenere informazioni sul processo di colatura e sulle tecniche di lavorazione applicate nel processo di produzione. Grazie all'elevato potere di penetrazione dei neutroni termici, le misure di carattere archeometrico eseguite tramite la diffrazione di neutroni permettono di determinare le proprietà interne del campione. In questo lavoro descriviamo la struttura dello strumento e presentiamo alcune misure eseguite come test preliminare su campioni standard in modo da poter mostrare l'elevato livello qualitativo dei dati ottenibili con questa macchina. In particolare ci soffermeremo su quei risultati che hanno rilevanza archeometrica quali, per esempio, analisi multi-fase di bronzi e determinazione a livello quantitativo delle impurezze.