Nondestructive Metallurgical Analysis of Astrolabes by Utilizing Synchrotron Radiation

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Introduction

The astrolabe was the most sophisticated instrument of pre-telescopic astronomy, and it was born out of man's curiosity with the night sky and desire to methodically map the stars' movement. It was used as a timepiece that could tell time during both the day and night, a surveying tool to measure distances and make more accurate maps, and a practical tool for all sorts of astronomical calculations.

Astrolabes represent the state-of-the-art in materials, design, and forming processes during their time of manufacture. As such, astrolabes are also a valuable instrument to be studied metallurgically and to learn about the technological history of man.

Because of their intricate engraving and the important historical place that astrolabes hold, they are highly desired by private collectors and museums alike. Historically, metallurgical analysis has been a destructive process requiring samples to be cut from the artifact, polished, and etched to reveal their microstructure and forming history. It follows that there has been very little metallurgical analysis [1] performed on astrolabes in the past, because collectors and curators do not want to have the instruments in their collections degraded in any manner.

One technique that is rare in the field of archaeometallurgy is the use of high-energy x-rays produced by a synchrotron. It is possible to perform diffraction experiments that give information about microstructure without damaging the object. It is also possible to obtain data on the chemical composition of the sample without requiring a sacrificial sample, as in emission spectroscopy experiments. Thus, studying rare and valuable astrolabes via synchrotron experiments allows analysis of the metallurgy of the astrolabes without damaging them [2-4]. A collection of more than 30 astrolabes has been examined, dating from 1250 A.D. to modern reproductions. Major production centers represented in this collection include Louvain (4) examples, Nuremberg (2), Lahore (6), and Isfahan (3).

Methods and Materials

Three main types of experiments were performed:

1. X-ray Diffraction Experiments. It is possible to determine the mechanical working history of the sample from the nature of its x-ray diffraction pattern. It is also possible to gain information about the bulk composition of the sample from the radial location of the rings in the x-ray diffraction pattern [5, 6].
2. X-ray Fluorescence Analysis. The near-surface composition of the sample can be obtained by measuring the secondary x-rays generated by the impinging x-ray beam [7]. This was performed to determine alloy compositions used for each astrolabe component.
3. X-ray Thickness Profiles. The transmitted intensity of the impinging x-ray beam is related to the thickness of the sample. Thus, by measuring the transmitted intensity, it is possible to determine the variation in thickness of the sample. Thickness profiles can provide information about the sample's forming history.

Results

From the collection of astrolabes studied, it was found that the subgroup of astrolabes from the Lahore region (dated 1601-1663 A.D.) showed brass alloys with a significantly higher zinc content than that of the rest of the astrolabes studied. Figure 1 illustrates this via the presence of β' in the x-ray diffraction results.

This figure is representative of the other astrolabe components from Lahore that were formed from brass sheet. The α-phase zinc composition of the brass astrolabes, as calculated from the diffraction patterns, is plotted versus the zinc composition measured by x-ray fluorescence in Fig. 2. It is seen that at higher zinc compositions, there is significant deviation between the two techniques.

The traditional method of brass production during the time of astrolabe use (900-1700 A.D.) was called the cementation method and limited the zinc composition to approximately 30 wt% [8]. This technique involves

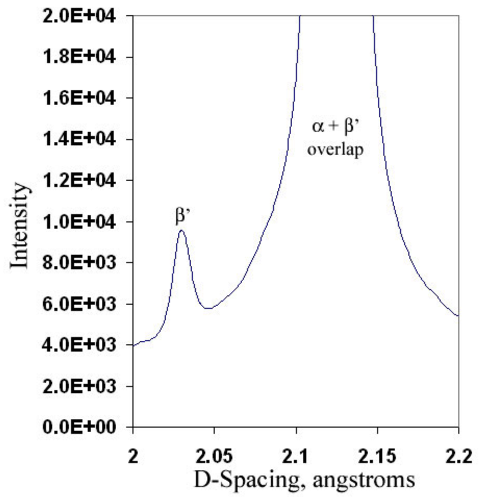

FIG. 1. Expanded region of diffraction peaks illustrating the presence of α and β' phases in astrolabe A-70.

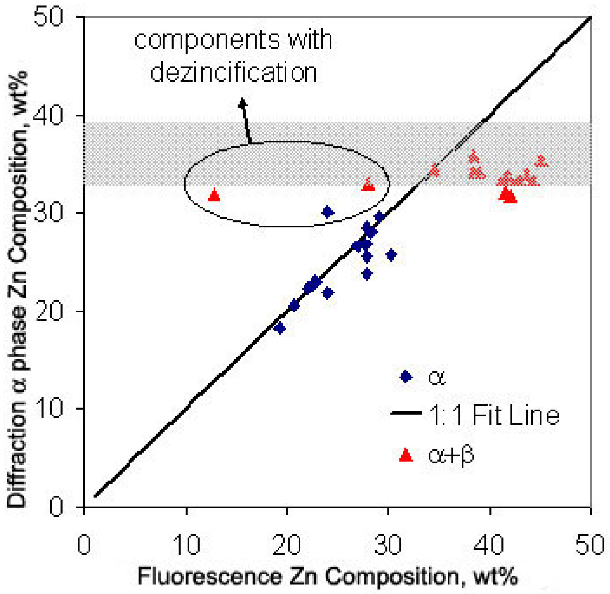

FIG. 2. Zinc composition in astrolabe components via diffraction and fluorescence experiments. The blue diamonds represent components of α brass only, while the orange triangles are $\alpha + \beta'$ brass.

reducing zinc oxide to zinc gas in the presence of copper pieces. The zinc gas diffuses into the copper to produce brass [8]. The astrolabes from Lahore show evidence of much higher zinc compositions by the presence of β′ (which does not appear until approximately 38 wt% zinc) in the microstructure, as shown in Fig. 1. Thus, the astrolabes must have been produced from brass manufactured by a different technique, most likely the direct co-melting metallic zinc and metallic copper.

It is known that zinc metal was first produced in large quantities in India during the 14th century, 400 years prior to production in Western Europe [9]. However, much of this zinc was reoxidized to form a very pure zinc oxide for medical uses as an eye salve [10]. While it has been suggested that some of the zinc was used for brass production [9], there has been no documented evidence for it. It is believed that these astrolabes from Lahore represent deliberate use of zinc metal to form brass.

In Fig. 2, there is a group of components that show evidence of significantly lower zinc compositions by x-ray fluorescence than by x-ray diffraction. Since the characteristic fluorescence x-rays for zinc (8.64 keV for K_α) have much lower energy than that of the primary beam energy (70-80 keV), they will escape only from the outer edges of the samples, even though they are generated throughout the beam’s interaction volume. Thus, one can consider the fluorescence composition as a “surface” composition and the diffraction composition as a “bulk” composition. For these three samples, the surface zinc composition was much lower than the bulk, which is believed to be evidence for de-zincification. This arises when brass is annealed in a zinc-free environment (such as open air); the zinc within the metal diffuses out and leaves a depleted layer around the surface. This can arise from annealing at too high a temperature or annealing for too long.

There is also a large difference on the right-hand side of the 1:1 line of Fig. 2 at high zinc compositions. The calculation for composition by diffraction is limited to single-phase binary alloys (α brass in this case). Once a second phase (β′ in this case) is introduced in the microstructure, the relative amounts of the phases change with composition instead of the lattice parameter of the single phase. This limit is represented by the gray band, which is the solubility limit for zinc in α brass. This is a band instead of a line due to the temperature dependence of the solubility. Thus, this limit will change with the annealing temperatures used when the brass is sheet-formed.

Discussion

The data give evidence for a localized region of advanced brass production technology centered around Lahore. The synchrotron provides critical access to data on the bulk “internal” structure and composition of the astrolabe samples. This is a necessity for studying samples that require completely nondestructive analysis.

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