scientifichighlights materials Skillful copper forging at the time of the Iceman

Neutron diffraction texture analysis tells how Ötzi and his contemporaries manufactured their copper axes. The proposed technique may become an important non-invasive tool in the characterisation of prehistoric metal objects.

The exceptional find of the 5300-year-old mummy of the Iceman and the study of the associated objects have revealed a wealth of information on the physical and cultural habits in the Copper Age (figure 1). The copper axe that was found associated with the frozen mummy, nicknamed Ötzi, found in 1991 in the Schnalstal glacier (Similaun, Western Alps) is the only known Copper-Age axe preserved with the original handle and bindings (figure 2). Within the frame of a project aimed at tracing the introduction of metallurgical techniques in the Alps and the development of metal work processes during the Late Neolithic and



Figure 1: Reconstruction of Ötzi clothing and appearance.

the Copper Age, a tentative experiment was made in order to employ a non-destructive technique to map the distribution of the copper crystallites in axe blades. Such prehistoric blades are rather rare and most museums are obviously reluctant to have them analysed by conventional invasive metallographic techniques.

Texture analysis is a powerful tool in the characterisation of polycrystalline materials. Specifically, texture studies performed by using penetrating neutron beams can be successfully employed in the investigation of cmthick objects such as the metal tools and the pieces of archaeological interest we are currently investigating. The texture study of metals has long been employed in the characterisation of metal objects produced by modern industrial processes. It is known for example that cast and melt-spun metals, cold-worked or cold-rolled metals, and metals worked through thermal



Figure 2: Ötzi copper axe bound to the original wooden handle made of *Taxus Baccata*.

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annealing cycles show rather different and well recognizable texture patterns, so that the texture maps observed on the final objects may readily yield information on the manufacturing process. These preliminary texture experiments were performed on the original Ötzi axe, and on other two coeval copper axes from the same area.

Neutron diffraction texture analysis experiments

The data have been collected at the D20 diffractometer, by using especially prepared supporting tools in order to ensure the integrity of the fine archaeological objects. Standard samples of copper prepared by simple casting or cold-working techniques were also measured as reference samples. The neutron powder diffraction data were collected from different areas of each axe and the texture analysis based on full-profile Rietveld procedures was carried out by using the MAUD software. The data were corrected for absorption by using both analytical and empirical descriptions of the axe's irregular shape. Examples of the resulting textures are described by the pole figures reported in figure 3.

The texture analysis immediately showed that copper crystallites in the standard as-cast samples are iso-oriented along the crystallographic [100] direction and produce a strong fibre texture (figure 3a). Some of the prehistoric axes such as the one from Castel-





Figure 3: Pole figures showing the different textures of copper crystallites resulting from different manufacturing techniques: (a) fibre texture produced by casting; (b) cube texture produced by cold working in the prehistoric axe from Castelrotto, and (c) absence of texture probably due to thermal annealing in the central part of the copper axe of Ötzi.

rotto (Bolzano, Italy) show the unequivocal features of the so-called "cube texture" (figure 3b), known in the modern metallographic literature as derived from cold-rolling of several metals, including copper and aluminum. The absence of the peculiar poles, that sometimes appear as satellites of the "cube" poles in modern cold-rolled metal sheets and are produced by the recrystallisation twin boundaries of the copper crystallites, indicates that the

3b

Castelrotto and similar axes have not undergone significant recrystallisation. Finally, the central portion of the Ötzi axe clearly shows no evidence of any of the previous textures, indicating extensive thermal annealing, probably due to several alternate cycles of cold working and thermal softening.

These preliminary results show that neutron diffraction texture analysis (NDTA) can be successfully employed to analyse the metallographic textures of bulky metal objects. Precious and rare prehistoric objects may be investigated without suffering any damage or mechanical stress. Neutron-induced radio activation in pure copper objects is limited to a few days, depending on the impurity content. The obtained texture maps go a long way in defining the metallurgical processes used to produce these important prehistoric tools and they are of great help in tracing the development of Alpine early metallurgy.