Conventional Hammering of a Fe-7.2%Ni Alloy Sample from Itutinga Meteorite: An Evaluation of Its Formability and Functionality

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Using a mechanical forming process used in the beginning of metallurgy, the samples from the iron meteorite Itutinga were heated and plastically deformed by hammering. Two sharp pieces, similar to small arrowheads, were produced and characterized microstructurally by optical microscopy and scanning electron microscopy (SEM/EDS/EBSD), complemented with Vickers hardness. As expected, the observations pointed to the presence of α2 phase, cracks, oxidation and deformed taenite lamellae. To assess the performance of the arrowheads a penetration test was done on a leather sample. The results demonstrated the possibility of making a useful rudimentary arrowhead from of iron meteorite.

Keywords: Itutinga, iron meteorite, hammering, taenite lamellae

INTRODUCTION

Iron meteorites were used by many human groups as a source of iron to produce artifacts. Over time, several tons have been used (Cooper, 2016) and a new material was included between those available, despite of its rarity, to produce tools and weapons, openning new possibilities for the creation of utensils.

Since most of the iron in the Earth exists in the form of oxides and the reduction of iron ores was not yet well known and practiced in the beginnings of Metallurgy, the use of metallics meteorites, as a source of iron, became attractive. Archaeologists have found in the cemetery of Gerzeh, in Egypt, beads of iron meteorite probably cold hammered dating from 3.300BC (Johnson, et.al., 2013). In the early days of Metallurgy heating of metals was done at temperatures between 800°C and 1300°C followed by hammering and rudimentary cooling procedures in air, water or sandbox in order to produce daily usefull artifacts (Serneels, 2016). This historical-cultural context allowed to stablish a vast field of interdisciplinar studies that is concerned with aspects regarding the production, use and consumption of metals since the first signs of metals industry – the Archeometallurgy (Killick and Fenn, 2012).

Once again with this work, in a first moment, it will be showed the possibility to make a primitive tool – similar to arrowheads – using rudimentary thermomechanical techniques as those available in the past. On the other hand, a microcharacterization of deformed structure provides information allowing to explain the plasticity of samples extracted from the Itutinga iron meteorite consisting of Fe-7.2%Ni alloy. In addition, with the arrowheads, we carried out penetration tests in a treated leather sample in order to access the performance of these tools for drilling.

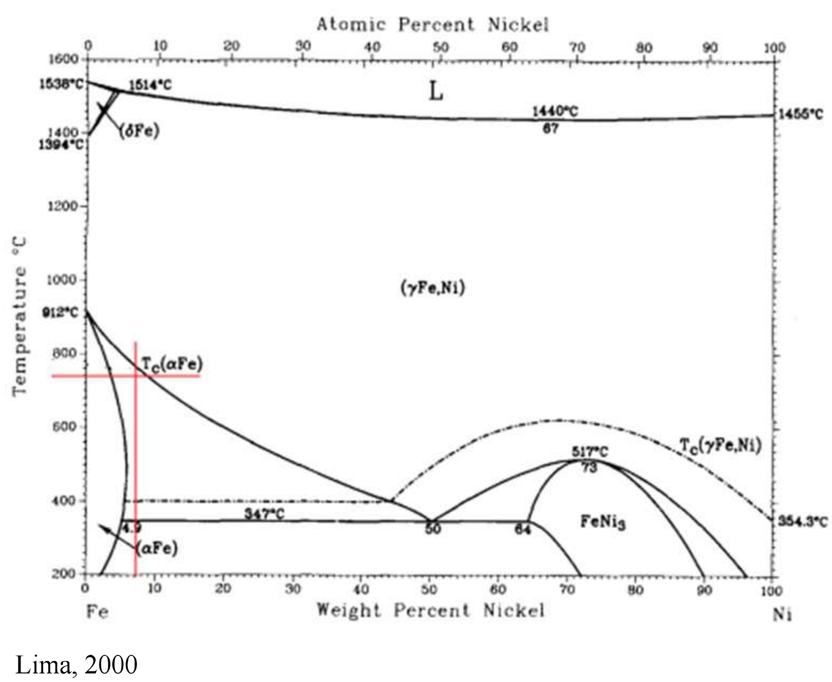
IRON METEORITES

Some crystallographic phases are found only in meteorites due to the very small diffusion coefficient of Ni into Fe on temperatures close to 500°C or less (Scorzelli, 1997). Published diffusion coefficients of Ni into Fe show that they are really small and decrease very fast with decreasing in temperature: 1 x 10 -20 m 2 /s at 600C; 1 x 10 -25 m 2 /s at 500C and 1 x 10 -31 m 2 /s at 300C. So, millions of years are necessary to complete diffusion processes at these temperatures. To prepare similar alloys in laboratory it is necessary to search for non-conventional methods, sometimes very complex (Lima, 2000).

According to the description due to (Swartzundruber, et.al., 1991) the equilibrium phases of the Fe-Ni system (Fig. 1) are: (1) the liquid L; (2) the b.c.c. high-temperature (δFe) solid solution; (3) the f.c.c. (ɤFe,Ni) solid solution; (4) the b.c.c., low-temperature (αFe) solid solution and (5) the FeNi3 intermetallic compound, which forms by a first-order order-disorder transformation below 517°C and has an extended range of homogeneity.

Considering this to the chemical composition of the Itutinga meteorite (Fe-7.2%Ni) one may expect (αFe) solid solution; and the f.c.c. (ɤFe, Ni) solid solution in its microstructure as shown in Figure 1.

FIGURE 1
Fe – Ni PHASES EQUILIBRIUM DIAGRAM



Lima, 2000

Meteorites are classified according to their chemical composition as Stony (basically minerals), Metallics (iron-nickel alloys in majority which structures have been formed in high pressured environments) and Metallic-Stony (iron and stony phases) (Zucolotto, et.al., 2013). The adopted chemical classification of iron meteorites is based on the content of traces elements as gallium and germanium besides the iron content (Buchwald, 1975). Instead of this, some phases are always present in iron meteorite; low nickel regions (kamacite) and lathes of high-nickel material (taenite). These phases belong to the well known Widmanstaten pattern. Structurally, iron meteorites are classified according to the average width of the lamellae of kamacite, revealed after suitable metallographic procedures (Woolfson, 1978). These two methods of classification – chemical and structural - present very good correlation (Sears, 1978). A content of Ni between 6 to 13% is peculiar to octahedrites, which consist of kamacite, taenite (ɤ-phase Ni-Fe, face-centred cubic lattice) and plessite (a fine intergrowth of kamacite and taenite) in varying proportions (Baldanza and Pialli, 1969). Fe-Ni meteorites provide a well-defined group of materials of fairly uniform composition. Iron-nickel meteorites exhibit a unique lamellar microstructure, a Widmanstaten structure, consisting of small regions with steep-iron-nickel composition gradientes (Christiansen, 1984) (Goldstein and Ogilvie, 1965). This microstructure is found in the Fe-Ni system only in meteorites, and is believed to arises as a result of slow cooling in a planetary core or other large mass (Hutchinson and Hagstrom, 2006). Meteorites with compositions consisting of between 5 and 17% nickel in iron are termed "octahedrite," and further characterized according to the width of the Ni-poor kamacite bands; "fine," (0.2-0.5 mm) "medium," (0.5-1.3 mm) and "coarse," (1.5-3.3 mm) (Benzerara et.al., 2002). Many meteorites have inclusions indicating that the material has been shocked at some point early in its evolution (Zucolotto, et.al., 2013). Following this the Itutinga meteorite is a medium octahedrite (Om) of the III AB group. It belongs to the collection of School of Mines Science and Techniques Museum of Federal University of Ouro Preto, in Minas Gerais State, Brazil. The main body of this meteorite weigths 46.21kg with mean kamacite lamellae width of 1.0±0.2mm (Buchwald, 1975), and average chemical composition of 7.2%Ni, 18.6ppm Gallium, 36.0ppm Germanium and 13.0ppm Iridium, Fe (balance) (Kracher, et.al., 1980).

MATERIALS AND METHODS

Material available for this work were samples extracted from the meteorite Itutinga. To establish a parallel with ancient process, close to archeometallurgy techniques two fragments of Itutinga meteorite were heated in a forge fed by coal, charcoal and blown by compressed air. The neighbourhoods of the samples attained temperatures in the range 740±10°C measured by a termocouple type k. After exposed for 20 minutes to this condition the fragments were shaped as small arrowheads by hammering in a conventional anvil. One may consider that these conditions resemble those of the begining of iron Metallurgy and they are not able to promote significant microstructural changes. Two cooling conditions have been chosen both close to ancient procedures: cooling in calm air and quenching in water at room temperature. In order to identify the phases present in these pieces, experimental analyses for microstructural characterization were carried out, basically optical and electron microscopy followed by Vickers hardness tests. For metallographic observations the arrowheads were send to surface preparation for examinations following the steps sanding, polishing and chemical etching with Nital (nitric acid diluted in 4% alcohol) as well as polishing with colloidal silica for EBSD analysis.

On the other hand to evaluate the ability of the tools (arrowheads) to be able to perform usefull daily functions they have been tested simulating a penetration action against a leather sample. The test leather plates had (7.0 x 7.0) cm by 4.0±0.5mm in thickness. Loads of 60kgf applied without impact and by free fall of the weight, travelling 5cm, were the two tests idealized to simulate the function of the arrowheads and to measure their ability to drill. Shore A hardness test of the leather revealed 88.8±4.0 SH.

RESULTS

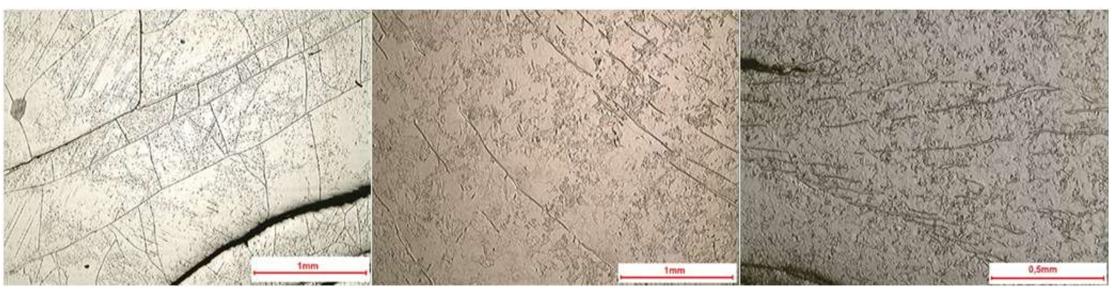
Following this methodology, after being heated treated in the forge the meteorite fragments were plastically deformed by hammering. At the end of the hammering process the arrowhead B was quenched (water at room temperature), while the other was cooled in calm air. Figure 2 (a,b,c) shows the forge (a), the hammering work (b) and the two finished arrowheads.

FIGURE 2
DETAILS OF THE WHOLE PROCESS TO OBTAIN THE ARROWHEADS: (A) THE FORGE (FIRE, THERMOCOUPLE, FRAGMENTS BEING HEATED); (B) HAMMERING; (C) ARROWHEADS A (AIR COOLED) AND B (QUENCHED)

Microstructural Observations

After hammering, the optical microscopy of the arrowhead A revealed oxidation of the edges and microcracks. The observation also identified α2 structure and deformed taenite lamellae due to the hammering process (Fig. 3 a, b, c).

FIGURE 3
OPTICAL MICROGRAPHIES: (A) MICROSTRUCTURE OF AN UNDEFORMED FRAGMENT OF THE METEORITE (40X); (B) TAENITE LAMELLAE AND α2 STRUCTURE IN ARROWHEAD A (40X); (C) TAENITE LAMELLAE AND α2 STRUCTURE IN ARROWHEAD B (80X)

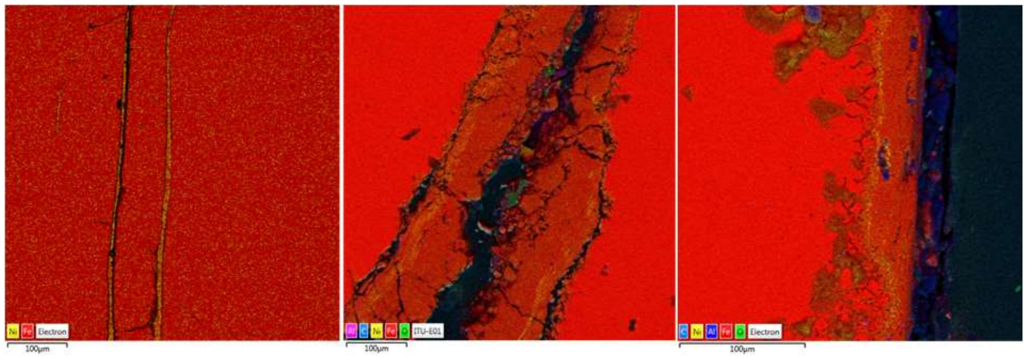


The α2 phase appears when the temperature sligthly exceeds 750°C, caused by the rapid undifferentiated transformation of the kamacite into a serrated and unbalanced phase (Buchwald, 1975). Thus, α2 may appears as a consequence of the heating-cooling cycle of the meteor during atmospheric transit; it may also come from controlled artificial treatment, as was observed (Kirsten, 1978). Arrowhead B heated at 740±10°C, conformed by the same way as A and quenched also revealed oxidation of the edges, microcracks, α2 structure as well as deformed and non-deformed taenite lamellae. The bending of the structures certainly reflects plastically deformed regions.

Energy Dispersive X-Ray Electronic Scanning (SEM/EDS)

SEM/EDS coupled with mapping distribution of chemical elements allowed to confirm the presence of kamacite and taenite in the fragments of the Itutinga meteorite. Also detected crack oxidation and the presence of the elements iron, nickel, aluminum and carbon (Fig. 4). Iron and Nickel are associated to phases αFeNi (kamacite), ɤFeNi (taenite); Oxigen to oxidation, carbon to sample inlay resin and aluminum to the abrasive paste for polishing. Oxidation in iron meteorites is favored even at relatively low temperatures in metallurgical terms, around 600°C (Buchwald, 1975). Oxidation will be more pronounced at temperatures above 800°C (Swartzundruber, et.al., 1991). SEM/EDS analysis of arrowhead B also revealed the same elements. Figure 4 (a,b,c) sumarizes the observations.

FIGURE 4
(A) UNDEFORMED FRAGMENT OF THE METEORITE (500X) (B) ARROWHEAD A: CRACKS, OXIDATION AND MAPPING DISTRIBUTION OF THE ELEMENTS (500X) (C) ARROWHEAD B: CRACKS, OXIDATION AND MAPPING DISTRIBUTION OF THE ELEMENTS (1000X)



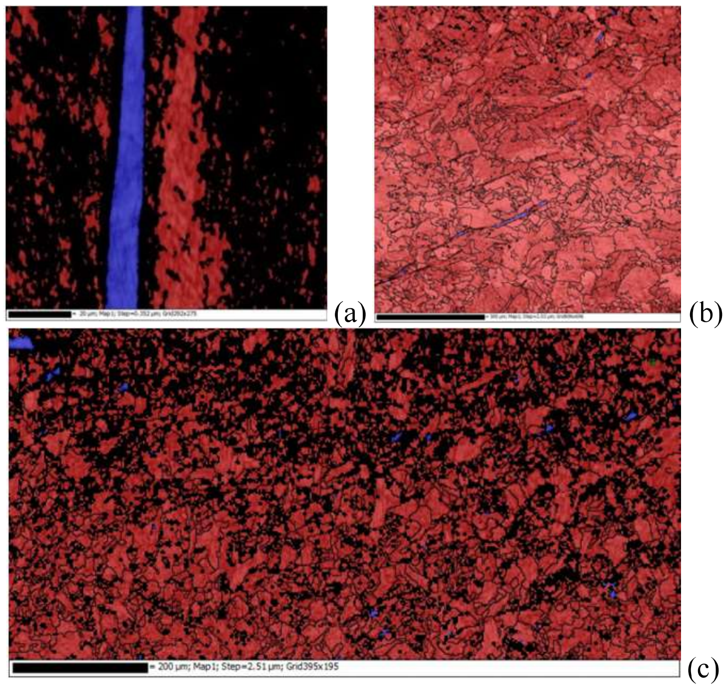
Vickers Hardness Test

In order to complement the microstructural characterization Vickers Hardness (VH) tests have been performed upon the arrowheads. In A (cooled in calm air at room temperature) 27 indentations and in B (water cooled) 15 indentations. Applied load 25 grams during 10 seconds for all tests. The average value of VH for the samples before thermal treatment, as extracted from de main body of the meteorite is 355.2±0.1VH. After heated treated and hammered A and B showed a decreasing in hardness: 220.4±0.1 for A and 257.9±0.1 for B, 38% and 27% decreasing respectively. According to the literature, the amount of phosphorus and nickel in the meteorite is responsible to the increase in VH value. Cosmic shock events promote a high initial hardness enabling kamacite to easily reach VH levels between 300 and 350 (Buchwald, 1975), compatible with the result found for the Itutinga meteorite. Further events such as cosmic annealing of highly shocked material followed by the passage through the atmosphere decrease surface hardness. Due to this uncontrolled thermomechanical events meteorites, in many cases, present very diferent hardness values reaching variations of about 100 units (Buchwald, 1975). As expected for some ferrous alloys the VH of the quenched piece is higher than that measured in the other arrowhead. In both cases this property is lower than the one found in the reference sample.

Electron BackScattering Diffraction (EBSD)

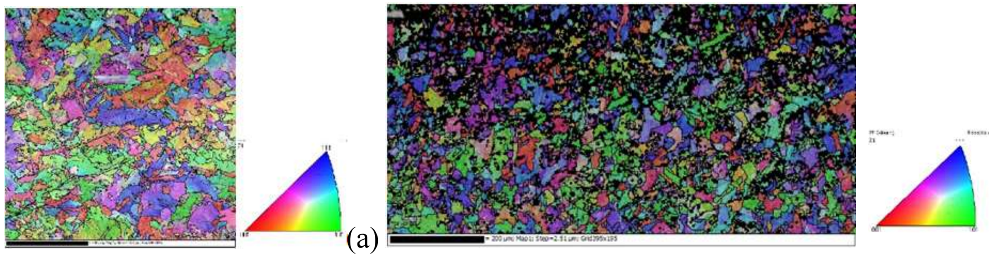
Electron Backscattering Diffraction data presents kamacite and taenite as indexed phases for all samples. At the undeformed fragment of the meteorite, well-defined laths of Taenite were identified in a matrix of Kamacite, Figure 5 (a). Poor-indexed diffraction patterns were also generated at this matrix due to the unsuitable metalographic sample preparation, somewhat that can also be due to the material itself. For its time, at the arrowheads, the EBSD indexed small amount of deformed Taenite phase surrounded by a Kamacite matrix, Figure 5 (b, c).

FIGURE 5
EBSD DATA PHASE MAPS SHOWING KAMACITE (RED), TAENITE (BLUE) AND UNIDENTIFIED PHASES, OR ZERO SOLUTION, (BLACK) IN THE SAMPLES:
(A) UNDEFORMED FRAGMENT OF THE METEORITE
(B) ARROWHEAD A (C) AND B



The crystallographic orientation data are shown by EBSD-collected orientations maps in Figure 6, in which colors are in correspondence with the inverse pole figures (IPF) of the cubic crystal system. In electron diffraction data, color variations mean local orientation changes, and deformed regions and grain boundaries usually produce diffraction patterns less well-defined and with lower band contrast values; then being represented always by the black color. The IPF-coloured orientation map collected for sample A shows different colors and also some grains. Inside these grains, one can note color variations, which is a representation of deformation bands in thermomechanically-worked materials Figure 6 (A). Unless for the lower grain size compared to sample A, the same observations can be made for sample B. For both samples, the EBSD-collected data points to no texture formation or preferred crystallographic orientation after worked, i.e., the thermomechanical processing applied on forging of each arrowhead was not able to rebuild the atomic structure of Kamacite and Taenite phases and just preliminary microstructural features such as deformation bands could be traced by electron diffraction; something completely understandable when the material worked is Fe-Ni solid solution phases without any impurity.

FIGURE 6
ORIENTATION MAPS FOR A AND B SAMPLES IN WHICH PREFERENCIAL ORIENTATIONS ARE NOT REVEALED

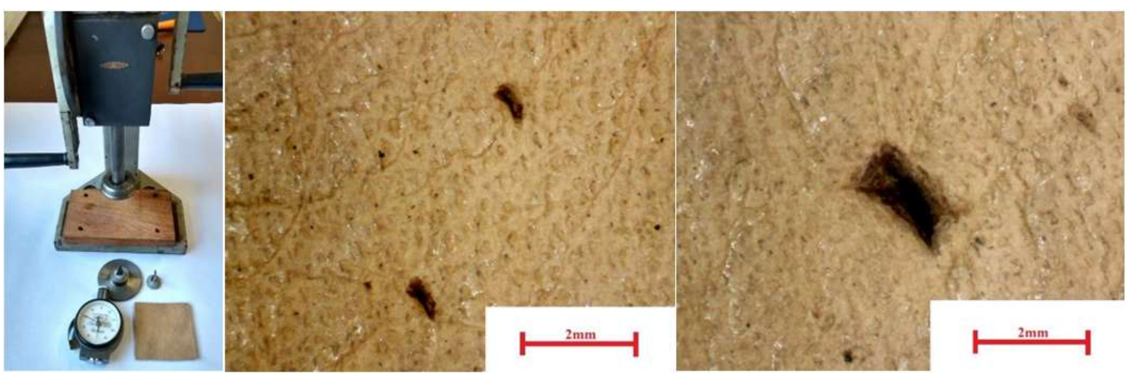


(a) (b)

Penetration Test

The performance of an arrowhead is directly related to its penetration capacity: the main function of this tool. In order to qualify this functionality penetration tests, against leather samples, were idealized and performed in laboratory. Two kinds of tests were conceived: static and dynamic. In both cases the mass available to press the arrowtips was 6kg. In the first type – static test - the tip of the arrowhead was carefully put in contact with de leather sample and the mass applied just by gravity action. On the contrary, for dynamic tests the mass fell from a heigh of 5cm. The arrowheads behaved very well during the tests without any damage. The dynamics test provided 2.94±0.02J kinetic energy for penetration into the leather. Figure 7 (a, b, c) shows the equipment to penetration test (a) and the indentations due to the drilling: in (b) for static test and in (c) for dynamic test.

FIGURE 7
(A) DRILLING EQUIPMENT (B) OPTICAL MICROGRAPHIES (40X) SHOWING TWO STATIC TEST INDENTATIONS (C) ONE DYNAMIC TEST INDENTATION (40X)



CONCLUSION

It was possible to forming two small arrowheads from Fe-7.2%Ni alloy fragments extracted from Itutinga meteorite using a conventional, not sophisticated, thermomechanical practice.

The microstructural observations confirm expected features of plastically deformed heat-treated metals/alloys: α2 phase, microcracks, deformed taenite lamellae and oxidation. Instead of plastic deformation no texture formation was detected.

Arrowheads A and B were sufficiently hard and though to penetrate a sample of treated leather. No damage was detected in the arrowtips.

Tools elaborated with the primordial metallurgical procedures exploited here could perform specific functions like drilling of leather. The possibility to deform plastically Fe-7.2%Ni alloys had been also demonstrated instead of the simplicity of the metalforming operation.

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