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AN ULTRASONIC "JACK HAMMER" FOR REMOVAL OF INCLUSIONS

by

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AN ULTRASONIC "JACK HAMMER" FOR REMOVAL OF INCLUSIONS

by

George L. Kehl,¹ Hyman Steinmetz,² and Warren J. McGonnagle³

In the identification of small inclusions in metals and alloys, it is customary to remove the inclusions of interest from an appropriate specimen (usually from a metallographic specimen) and to subject a finely divided sample to appropriate analysis. Techniques of removal consist of either chemical or mechanical methods. In the former, reagents are employed that are selective in their dissolution property, i.e., they will selectively attack the matrix metal and leave the included material as loose debris which may be filtered subsequently from the reagent and gathered for analysis. The principal disadvantages of this method are the uncertainties that the inclusion debris has not suffered some chemical change and that the residue consists of just the inclusions of interest and not an agglomerate of different kinds.

Removal of inclusions by mechanical methods has been confined largely to probing and loosening inclusions from the matrix metal with a pointed stylus. The operations are generally carried out under a microscope and the probe manipulated by hand. The method is time consuming and, because of awkwardness of the operations, it is often tedious to secure a sufficient sample for analysis.

When inclusions are large and polycrystalline in character, or small and segregated in mass, X-ray diffraction techniques often permit identification without removal. The techniques, however, are sometimes difficult to execute, and the success in identification depends in part upon the size of the collimated X-ray beam, and the precision of incidence on the region containing the specimen.

This paper describes a mechanical method for removing inclusions of a minimum diameter of about 10 microns by means of a pointed stylus oscillating at ultrasonic frequencies. The method works admirably well in

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removing hard and brittle secondary alloy phases, and nonmetallic inclusions of either the hard, glassy type or the loose, fluffy kind. Relatively soft and ductile secondary phases are not removed or shattered; rather, they are merely cold worked in situ. This circumstance, however, may be of interest in critical recrystallization studies where it is desired to work harden a selected single grain within a polycrystalline aggregate.

The extraction apparatus to be described consists of the following major components:⁴ (1) an ultrasonic driving oscillator designed to match the frequency of a selected barium titanate transducer, (2) a metallic horn attached to the transducer to magnify linear motion, (3) a pointed stylus that makes contact with the inclusion to be removed, (4) a bench-type metallurgical microscope, and (5) two micropositioners, one to hold the transducer and horn assembly and to permit locating the pointed stylus with precision, and the other to manipulate a quartz fiber to gather up the inclusion debris after extraction.

Ultrasonic Generator

The ultrasonic oscillator, diagrammatically shown in Figure 1, is designed to match the natural frequency of a selected barium titanate transducer (47 kc).⁵ The circuit of the generator is conventional, except for the incorporation of a feed-back amplifier system to secure constant amplitude. The unit is capable of delivering energy well in excess of that required for the intended purpose.

Transducer-Horn Assembly

The transducer assembly, illustrated in Figure 2, consists of the barium titanate transducer and an attached brass horn. The two component parts are cemented together with Tuffbond or with a similar adhesive. The purpose of the horn⁶ is to serve as a transformer and to increase the linear motion of the stylus relative to that of the transducer by a factor of about 30. In the horn design to be described, the throw of the stylus tip is about 0.001 in. each way from center.

⁴The complete extraction apparatus is commercially available on order from C. A. Brinkman and Co., Great Neck, Long Island, New York.

⁵Available from Gulton Manufacturing Co., Metuchen, New Jersey, and Brush Electronics Co., Cleveland, Ohio.

⁶Mason, W. P. and Wick, R. F., "A Barium Titanate Transducer Capable of Large Motion at Ultrasonic Frequency," J. of Acoustical Soc. of Am., Vol. 23, 1951.

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To secure maximum transfer of energy to the stylus, two factors must be considered in the design of the horn. The diameter of the base should be less than one-half of the wave length of the ultrasonic wave passing through the horn material, and the length of the horn from the base to the tip of the stylus should be equivalent to an integral number of half wave lengths. When this latter requirement is fully realized, the cemented interface of the assembly will be at a pressure node with attendant least stress on the joint, and the stylus tip will vibrate with maximum amplitude.

For a transducer of given resonance frequency, the wave length and physical size of the horn will vary depending upon the material from which the horn is constructed. From the relationship,

$$V = f\lambda \quad , \quad (1)$$

where V = velocity of sound through the horn material, in. per sec
(for single phase brass, 174,000 in. per sec),

f = frequency of transducer, cycles per sec,

λ = wave length, in.,

the wave length may be calculated and the maximum diameter of the horn base determined.

The taper of the horn, from the base to the small end, is best designed to be of an exponential shape following the familiar mathematical expression,

$$A = A_1 e^{cx} \quad ,$$

where A = cross-sectional area at any distance x from the large end

A_1 = area at large end

c = constant.

In order to avoid an excessively long horn tip, slight departure from an exponential taper can be tolerated without seriously affecting performance. Machining of the taper is facilitated by matching the contour against a pasteboard template.

To facilitate change of the stylus, which is waxed to the small end of the taper in a manner to be described, the lower portion of the horn is detachable from the upper end by screw threads, as illustrated in Figure 2b. This arrangement permits a number of extra horn ends to be fitted with styli and to serve as ready replacements when points are damaged in use.

Preparation of Styli

At the onset of this development, styli were made from 0.02-in. diameter tungsten wire, electrolytically pointed by a method first described by Pfann.⁷ For the application intended, tungsten is not entirely satisfactory owing to its relative softness, short life as a tool, and the loss of tungsten during use which will contaminate the inclusion debris.

Currently, fully hardened, high carbon steel styli are being used. Forty-mil diameter wire, after heat treatment, is electrolytically pointed in an electrolyte consisting of 50% nitric acid - 50% glacial acetic acid, cooled in an ice bath during use. The wire serves as one electrode of the cell and a strip of platinum foil as the other. The wire is suspended from the platform of a micropositioner into the electrolyte to a depth of about 1/8 in. below the reagent surface.

By application of 8 to 12 volts (AC) across the cell, the immersed section of the wire becomes uniformly reduced in cross section with an attendant decrease in current flow. The wire is raised slowly out of the electrolyte just before the current reaches zero. This final operation is somewhat critical but can be successfully executed after a few trials. The time required is about one minute. Figure 3 illustrates the point formed by the prescribed method.

The pointed stylus is cut to proper length (the order of 1/2 in.), to fulfill the requirement of total horn length, and is subsequently placed into a 0.043-in. diameter hole at the end of the horn, being affixed in position with softened hard wax.

Extraction Procedure

The arrangement of the component parts of the apparatus are shown in Figure 4, with the exception that the micropositioner supporting the pick-up fiber is normally placed to the right of the microscope. A specially designed glass hood is placed over the specimen, as illustrated, and purified argon gas passed into the system during the extraction operation. A slit in the hood is provided to allow entry of the stylus into the chamber. This hooding arrangement is necessary if the frictional heat generated at the stylus tip is sufficient to cause air oxidation of the inclusion debris, or if significant oxidation occurs because of the fineness of particle size of the extracted material. However, if the debris of interest does not suffer oxidation during extraction, the hood may be removed and the refracting objective replaced by a reflector to provide greater working space above the specimen surface.

⁷Pfann, W. G., "An Electrolytic Method for Pointing Tungsten Wires," Trans. AIME, Vol. 175, 1948.

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The area of interest on the specimen is located microscopically and, while observing the operation, the vibrating stylus is lowered until the point makes contact with the inclusion. When this occurs, the inclusion is shattered locally and a shower of debris is formed and scattered in the vicinity. The depth of penetration and the size of the resulting hole in the inclusion are readily controlled by manipulation of the stylus through actuation of the micropositioner.

When sufficient inclusion debris has been created⁸ to serve as an X-ray diffraction specimen, the fragments are gathered on a glass or quartz fiber suitably coated with grease or an adhesive such as Vistanex. The fiber is merely moved over the specimen surface by means of a micropositioner and contact made with the loose debris.

Figures 5 to 8 are illustrative examples of inclusions partially removed by the method described. The inclusion debris is self-evident, and the precision of the extraction method is noted by the position of the resulting cavities within the inclusions.

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⁸In most cases this may be accomplished in 15 - 20 minutes, excluding the time required to position the specimen.

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Wiring Diagram of the Ultrasonic Generator.

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A

a. Component parts assembled

B

b. Component parts disassembled

Figure 2

The Barium Titanate Transducer and Horn Assembly, but
Without Stylus Attached.

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Figure 3

Representative Shape of Steel Stylus Point as
Electrolytically Formed. 25X

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Figure 4

Illustrating the Microscope Arrangement and
Transducer Assembly in Operating Position.

Figure 5

Inclusion of UC in a Uranium Matrix. Note scattered inclusion debris and cavities in inclusions resulting from the vibrating stylus. Unetched. 150X

Figure 6

A Uranium Matrix Containing Inclusions of UO_2 (Dark) and $U(C, N, O)$ (Light). Note evidence of precision in positioning stylus on the various inclusions of interest. Unetched. 150X

Figure 7

Copper Oxide Phase in a Matrix of Copper. Note areas in the primary phase where material has been removed. Unetched. 250X

Figure 8

Graphite Flakes in Cast Iron Partially Removed from the Matrix. Unetched. 250X