27
3/14/78
25-D-NT-LS

SAND78-0144
Unlimited Release

3151

ULTRASONIC ATTENUATION AS A FUNCTION OF
HEAT TREATMENT AND GRAIN SIZE IN 79Ni-6Mo-15Fe ALLOY

MASTER

John H. Gieske

Sandia Laboratories

IF 2900 Q(7-73)

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

ULTRASONIC ATTENUATION AS A FUNCTION OF
HEAT TREATMENT AND GRAIN SIZE IN 79Ni-6Mo-15Fe ALLOY

John H. Gieske
NDT Technology Division 9352
Sandia Laboratories
Albuquerque, New Mexico 87115

ABSTRACT

A pulse echo ultrasonic technique was used to measure the attenuation coefficient for 79Ni-6Mo-15Fe alloy specimens. The attenuation coefficient was determined using a 25 MHz ultrasonic transducer for specimens which had undergone different time-temperature heat treatments. The ultrasonic attenuation data versus heat treat time was used to assess grain size growth in the specimens.

Printed in the United States of America

Available from
National Technical Information Service
U. S. Department of Commerce
5285 Port Royal Road
Springfield, VA 22161

Price: Printed Copy \$4.00; Microfiche \$3.00

NOTICE
This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

ACKNOWLEDGMENT

thor wishes to thank M. M. Karnowsky, 5832, for proposing that
transonic measurements be made and for supplying the heat treated
ins.

INTRODUCTION

A study is being made of the 79Ni-6Mo-15Fe alloy to determine its appropriateness as an anti-intrusion cable. In the course of this study a fast nondestructive technique for measuring the average grain size of the alloy was sought in order to determine the effects of annealing temperature and time on the growth of the grains in the alloy. Ultrasonic attenuation techniques have been successfully applied in the determination of grain size and microstructure composition for a number of metals and alloys^{1,2,3,4,5,6}. Therefore, ultrasonic attenuation was chosen as a method to measure the effective or average grain size differences between the various annealed specimens of the 79Ni-6Mo-15Fe alloy. If a correlation between average grain size and ultrasonic attenuation is established for the alloy, then the ultrasonic attenuation measurement can be used to assure that a suitable grain structure has been achieved by the proper temperature-time anneal of the alloy.

The ultrasonic attenuation coefficient is defined as the logarithmic ratio between the incident and attenuated ultrasonic energies I_0 and I as

$$\alpha = \ln I_0 / I. \quad (1)$$

Due to the scattering of the ultrasonic wave by the microstructure of the medium, α can be related to the average grain diameter D and the frequency f of the ultrasonic wave for various dimensional conditions of the ultrasonic wavelength λ . There are three ultrasonic attenuation formulae which depend on the λ/D ratio:

1. Rayleigh scattering region $\lambda > 3D$

$$\alpha = A_1 D^3 f^4 \quad (2)$$

2. Stochastic scattering region $\lambda \leq D$

$$\alpha = A_2 D f^2 \quad (3)$$

3. Diffusion-absorption region $\lambda < D$

$$\alpha = A_3/D + A_4 f + A_5 f^2 \quad (4)$$

where $A_1, A_2, \dots$ are constants which depend on the density and the elastic anisotropy between the individual grains.

Generally for most engineering materials the attenuation coefficient does not depend on the frequency by the exact exponents as given in the previous equations. This is especially true where a large variation in microstructure exists in small regions of the material. However, one may establish an empirical relationship between α and f for a given material⁵ which can be correlated with the average grain size D . In the same way one may also establish an empirical relationship between α and D for a constant frequency. If D depends on other parameters such as annealing temperature and time, then α versus time for a constant temperature can be used as a measurement of average grain size growth. It is this last relationship which was used in assessing the effects of heat treatment on the average grain size growth in the 79Ni-6Mo-Fe alloy.

The same relationship between the average grain size versus heat treatment time at a constant temperature may be obtained by measuring grain sizes from photomicrographs. The procedure is straightforward for uniform grain size structures but the procedure is quite tedious and unreliable where large nonuniform distributions of grain size may be present. The grain size distribution in the 79Ni-6Mo-15Fe alloy is of the latter case. The advantage of the ultrasonic technique is that an effective or average grain size determination is made from a

single measurement since the ultrasonic beam width is at least an order of magnitude larger than the individual grains and it travels through twice the full thickness of the specimen. There is also no special preparation of the specimen surface for the ultrasonic measurement which is required for taking a photomicrograph.

EXPERIMENTAL PROCEDURE

The 79Ni-64Co-15Fe alloy specimens were standard size strips of approximately 1.27cm width, 0.05cm thickness, and 28cm length. The material was acquired commercially from three different suppliers; viz, Allegheny Ludlum (A), Carpenter Technology Corporation (C), and Magnetics Incorporated (M). The thickness dimension of the strip varied slightly between suppliers where values of 0.0508cm (A), 0.0643cm (C), and 0.0495cm (M) were measured respectively. The method of preparation of the material for the first two suppliers (A and C) was a vacuum melting procedure whereas for the third supplier (M) it was of a powder metallurgy procedure.

Since each supplier used a different process in preparing the 79Ni-64Co-15Fe strips, one specimen from each supplier was used for each time-temperature heat treat sequence. The ultrasonic attenuation measurements were made in the thickness direction of the strips near the center of each specimen.

The ultrasonic measurements were made by using a water immersion pulse echo technique using a 25 MHz 1/4 inch diameter damped transducer. A block diagram of the technique for recording the ultrasonic attenuation data is shown in Figure 1. Examples of the X-Y recorded traces of the ultrasonic echoes for specimens from each supplier in the as received condition are shown in Figure 2 and for specimens aged for 16 hours at 1050° F are shown in Figure 3.

As indicated in Figure 2, the amplitude ratio $B = b_{n+1}/b_n$ of any two consecutive echo peaks determines a measurement of the attenuation coefficient at a given frequency by the expression

$$\alpha = \{ \ln(R/B) / 2d \} \quad (5)$$

where d is the thickness of the specimen and R is the water-to-specimen interface reflection coefficient which is given by

$$R = (\rho V - \rho_w V_w) / (\rho V + \rho_w V_w). \quad (6)$$

The density ρ of the the specimen alloy was measured to be 8.66 g/cc and the ultrasonic velocity V in the specimen was measured to be 0.576 cm/ μ sec. Using the value of 0.143 g/cm²/ μ sec for $\rho_w V_w$ of water gives a value of 0.944 for R . The value $B = b_{n+1}/b_n$ was calculated for as many pairs of echoes as reasonable from the recorded X-Y trace. An average value of B was then obtained and used for the calculation of the attenuation coefficient α . The first pair of echoes was not used in any of the calculations since they may be influenced by the ring down of the front surface echo.

RESULTS AND DISCUSSION

The calculated values of the attenuation coefficient for each specimen for the 25 Mhz frequency are listed in Table 1. The data of Table 1 is presented in Figures 4, 5, and 6 where α versus time in hours is plotted for each constant temperature sequence. A least squares linear fit to the data in the portion of the data that appears to be linear with time is also plotted in the Figures. The linear fit condition is justified since the wavelength of the ultrasonic wave in

the heat treated material is $\lambda = V/f = 0.576/25.0\text{cm} = 0.023\text{cm}$, which is in the range of the average grain size as measured from several photomicrographs. Therefore α should be proportional to D as expressed in Equation (3).

As seen in Figure 4, for the M specimens, the dependence of α on time indicates therefore that D is increasing with time as well as increasing at a greater rate at the higher temperatures. From Figure 4 it is also suggested that a final attenuation coefficient of approximately 0.36neper/mm or slightly higher will be reached for all temperature time sequences.

The data of Figures 5 and 6, however, suggest that very little grain size growth is occurring in the A and C specimens after the initial short time temperature heat treatment. Therefore, only one least square linear fit for all the data of each Figure was made and the resulting curve is shown in each Figure. For both the A and C specimens it appears that the final attenuation coefficient of approximately 0.36neper/mm will be reached for all temperature time sequences which agrees with the final value reached with the M specimens. The scatter of the data exhibited in Figure 6 for the C specimens is comparatively smaller than that of Figure 5 for the A specimens. This is thought to be due to the fact that the C specimens were 5% thicker which provided a more reliable measurement.

In conclusion, the attenuation data of Figures 5 and 6 suggest that for both the A and C specimens only a short heat treatment at 1050°F is necessary to achieve the desired grain structure whereas from the attenuation data of Figure 4 for the M specimens only a relatively short heat treatment at 1200°F will achieve the same effect.

FIGURE 1
BLOCK DIAGRAM OF RECORDING
ULTRASONIC ATTENUATION DATA

FIGURE 2
 ULTRASONIC PULSE ECHO TRACES FOR AS RECEIVED
 SPECIMENS
 A - Allegheny Ludlum
 C - Carpenter Technology Corp.
 M - Magnetics Incorp.

FIGURE 3
ULTRASONIC PULSE ECHO TRACES FOR SPECIMENS
HEAT TREATED AT 1050° F FOR 16 HOURS

FIGURE 4

ATTENUATION COEFFICIENT VERSUS HEAT TREATMENT TIME
FOR MAGNETICS INCORPORATED SPECIMENS (M)

FIGURE 5

ATTENUATION COEFFICIENT VERSUS HEAT TREATMENT TIME
FOR ALLEGHENY LUDLUM SPECIMENS (A)

FIGURE 6

ATTENUATION COEFFICIENT VERSUS HEAT TREATMENT TIME
FOR CARPENTER TECHNOLOGY CORPORATION SPECIMENS (C)

TABLE 1

ULTRASONIC ATTENUATION COEFFICIENT α IN
neper/mm FOR TEMPERATURE TIME SEQUENCES

A - ALLEGHENY ALUMINUM SPECIMEN
C - CARPENTER TECHNOLOGY CORP. SPECIMEN
M - MAGNETICS INCORP. SPECIMEN

Temperature & Specimen	Attenuation Coefficient for Heat Treat Time in Hours							
	<u>0</u>	<u>1</u>	<u>2</u>	<u>4</u>	<u>8</u>	<u>16</u>	<u>24</u>	<u>48</u>
1050°F								
A	.058	----	----	.497	.353	.353	.353	.353
C	.020	----	----	.340	.327	.407	.353	.366
M	.037	----	----	.167	.246	.274	.302	.346
1100°F								
A	.058	----	.240	.227	.439	.323	.368	----
C	.020	----	.303	.393	.353	.366	.315	----
M	.037	----	.219	.193	.288	.288	.302	----
1150°F								
A	.058	.309	.295	.253	.368	.227	----	----
C	.020	.379	.379	.309	.327	.291	----	----
M	.037	.180	.260	.246	.260	.331	----	----
1200°F								
A	.058	.338	.338	.414	.253	----	----	----
C	.020	.303	.291	.407	.279	----	----	----
M	.037	.246	.317	.288	.346	----	----	----

REFERENCES

1. DiGiacomo, G.; Ohlstein, G.; and Jones, W. J., "Development of an Ultrasonic Method for Determination of Grain Size in Cast Steel," Materials Evaluation, December 1970, 271-276.
2. Roman, I.; Oron, M.; Pesach, J.; and Rosen, M., "Determination of Grain Size in Uranium-Chromium Alloys by Ultrasonic Attenuation," J. Mat. Sci. 9, (1974), 630-634.
3. Roderick, R. L. and Truell, R., "The Measurement of Ultrasonic Attenuation in Solids by the Pulse Technique and Some Results in Steel," J. Appl. Phys. 23, No. 2 (1952), 267-279.
4. Roth, W., "Scattering of Ultrasonic Radiation in Polycrystalline Metals," J. Appl. Phys. 19 Oct. (1948), 901-910.
5. Vary, A., "The Feasibility of Ranking Material Fracture Toughness by Ultrasonic Attenuation Measurements," J. Testing and Evaluation, 4, No. 4, July 1976, 251-256.
6. Mason, W. P., and McSkimin, H. J., "Energy Losses of Sound Waves in Metals Due to Scattering and Diffusion," J. Appl. Phys., 19, October (1948), 940.