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OVERLAY COPY TECHNIQUE TO PROVIDE HIGH-CONTRAST ELECTRON MICROGRAPHS FOR AUTOMATIC METALLOGRAPHIC ANALYSIS

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SUMMARY

The use of automatic film scanning instruments for determining quantitative microstructural parameters of multiphase specimens examined by electron microscopy has been severely handicapped by the difficulty in obtaining an image of the microstructure with sufficient contrast between phases. An overlay copy technique with an automatic film scanner to obtain the required contrast is described. The technique is illustrated by determining particle-size distribution and volume-percent dispersoid in a specimen of nickel containing thoria particles. Results are in good agreement with those from a commercial semiautomatic particle-size analyzer. The overlay copy technique has general application to metallographic analysis of multiphase microstructures with various measurement methods.

INTRODUCTION

Automatic film scanning instruments are of interest as a means of rapidly determining quantitative microstructural parameters in metallic and ceramic multiphase specimens (ref. 1). The use of such instruments for analyzing electron micrographs prepared from replicas, however, has been severely handicapped by the difficulty in obtaining a photographic image of the microstructure with sufficient contrast between phases to give accurate results. Refinements in shadowing techniques for electron microscopy have improved definition of interphase boundaries only. Improvements in replica preparation and in photographic techniques have not, as yet, provided sufficient contrast between phases, for example, between the particles and the matrix in a dispersion microstructure. In addition, direct imaging of the microstructure does not, in general, permit selection of the phases to be measured or exclusion of pores, unwanted phases, or artifacts. A

method for obtaining the necessary phase contrast in electron micrographs made with replication techniques is described herein. This method permits measurement of phases with an automatic film scanner and the preselection of the described phases or structures to be measured. The technique is illustrated by determining particle-size distribution and volume-percent dispersoid in a sample of TD-Nickel (thoria particles in a nickel matrix). Measurements with an automatic film scanner were compared with those made with a commercial semiautomatic particle-size analyzer, which previously had given reasonably accurate results.

MATERIALS, APPARATUS, AND PROCEDURE

The TD-Nickel examined, nominally Nickel + 2-weight-percent thoria, is an early version of that product and shows a coarser dispersion than is currently being produced. A specimen was cut from a bar and annealed at 1100° C for 1 hour in vacuum. A transverse section was examined.

Apparatus

The automatic film scanner was a Flying Spot Particle Quantizer and Lineal Analyzer manufactured by the Airborne Instrument Laboratory of Cutler-Hammer (ref. 2) for the Lewis Research Center. This instrument was specifically designed to obtain parameters of dispersion microstructures. In the instrument a cathode-ray tube scans a raster (linear scan pattern) on the 35-millimeter film being examined. A photomultiplier tube, which detects variations in intensity of the light spot after it has passed through the film and a high-resolution optical system, feeds logic circuits and a monitor oscilloscope. The output from the logic circuits is fed to a digital display counter. In this study, the instrument measured total scan line across the matrix and total scan line across the particles. It also counted individual particles and sized them in accordance with their maximum intercept length.

A Zeiss Particle Size Analyzer was used to count and to size dispersoid particles for comparison purposes. In this semiautomatic instrument an iris diaphragm is used to control the diameter of an illuminated circular spot, which is projected onto a photographic print. The area of the lighted spot is matched visually by the operator to the area of a particle. When the particle is matched, the operator closes a switch; this procedure automatically classifies that particle into one of 48 size ranges and records it as a unit count on the display counter corresponding to that size range. Particles were therefore counted and sized according to their number and area in the micrograph. The use of this apparatus for the measurement of particle size is reported in reference 3.

Procedure

The specimen of TD-Nickel was mounted, polished, etched, and replicated according to standard procedures for electron microscopy. One photographic print at a magnification of 45 000 was used for analysis.

An overlay copy technique for preparing a high-contrast 35-millimeter film for the automatic film scanner was used and consisted of the following four steps:

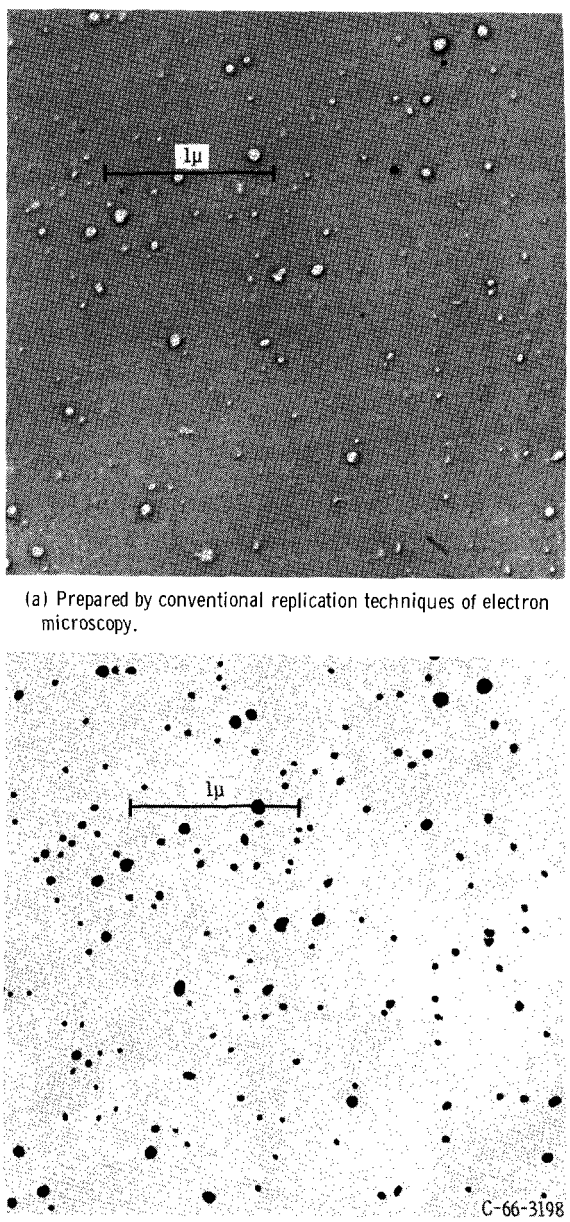
(1) A photographic print of the microstructure (fig. 1(a)) was stapled below a transparent plastic sheet.

(2) The thoria particle images to be measured were blackened on the plastic sheet with either a crayon designed for writing on glazed surfaces or a pen containing an ink or paint which did not spread on the plastic. The limits of the micrograph field were also marked to maintain the correct field area.

(3) After marking, the plastic sheet was trimmed to the size of the field in the micrograph and taped to a sheet of white paper.

(4) The black-on-white subject was rephotographed to give a high-contrast 35-millimeter film negative suitable for use in the film scanner. The film negative was then positioned in the film scanner, and particle-size distribution and particle and matrix traversed lengths were measured.

The values derived from the use of the automatic film scanner were compared with similar values for the same area of the specimen made with a Zeiss semiautomatic particle-size analyzer. To permit examination of the same area, a photographic print (at a magnification of 45 000) was made from the same 35-millimeter film prepared for the film scanner. This print (fig. 1(b)) was used with the particle-size analyzer so that



(a) Prepared by conventional replication techniques of electron microscopy.

(b) Printed from film prepared by overlay copy technique.

Figure 1. - Micrographs of TD-Nickel prepared by conventional electron microscopy and by overlay technique. Original magnification, 45 000; reduced 49 percent in printing.

the same enhanced image was measured in both instruments. Exploring the absolute accuracies of either method of analysis was beyond the scope of this investigation, and only a relative comparison of the two methods was made.

The particle-size distributions were determined with both instruments by correcting particle diameters on the film or the micrograph by the appropriate magnification factors. The volume percentage of dispersoid was obtained in the film scanner from the usual lineal analysis relation for volume fraction, namely, the quotient $L_a/(L_a + L_m)$, where L_a is the total scan line across the particles and L_m is the total scan line across the matrix. For the Zeiss particle-size analyzer, the sum of the individual particle areas was divided by the total micrograph area.

RESULTS AND DISCUSSION

The results of this study have demonstrated the feasibility of using a transparent-overlay copy technique for producing high-contrast images of dispersion microstructures suitable for analysis in an automatic film scanner. The achievement of high contrast between the matrix and the dispersoid is illustrated in figure 1. Figure 1(a) is the micrograph of the TD-Nickel studied, showing the fine thoria particles to be counted and sized, while figure 1(b) is a print made from the 35-millimeter film prepared by the overlay technique.

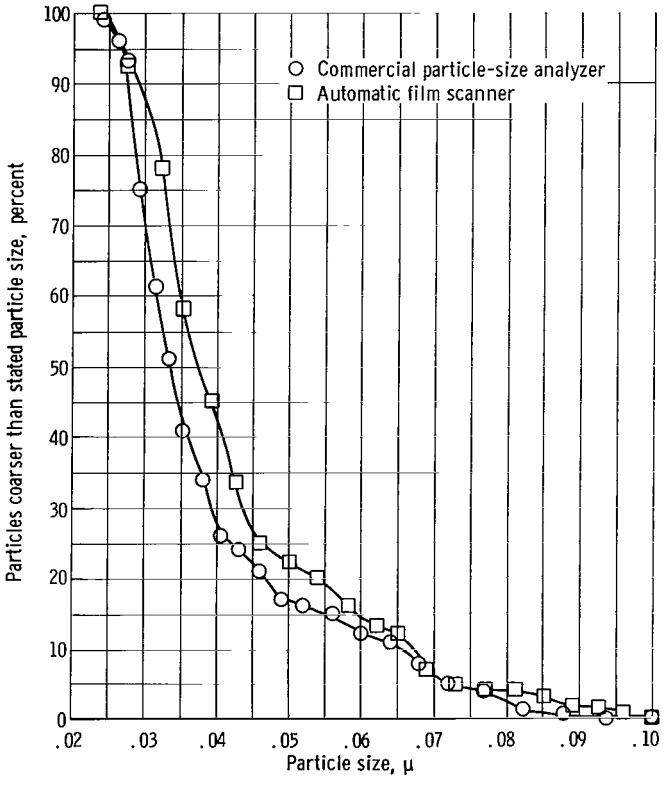


Figure 2. - Measurements of particle-size distributions in TD-Nickel by two methods of analysis.

The results of determinations of particle-size distribution and volume-percent dispersoid for the same area of the specimen by the two methods of analysis are given in figure 2 and table I. Figure 2 shows the cumulative distribution of thoria particle sizes. The curve obtained with the automatic film scanner indicates a somewhat coarser particle distribution than that obtained with the commercial particle-size analyzer. The displacement averages approximately 10 percent of the (larger) value obtained with the automatic film scanner and is less at the extremes of the particle-size range.

TABLE I. - MEASUREMENT OF AMOUNT OF DISPERSOID IN TD-NICKEL BY TWO METHODS OF ANALYSIS

[Number of particles counted, 161.]

Method of Analysis	Measured amount of dispersoid, volume percent
Automatic film scanner	1.92
Commercial particle-size analyzer	1.88

This discrepancy is believed to be due to differences in the definition of particle size by the two methods. In the automatic film scanner, particle size is the maximum intercept along the scan direction, while with the Zeiss instrument it is the diameter of an equivalent area circle. These values would not be expected to be identical for irregular particles.

Values for the measured volume-percent dispersoid are given in table I. The value obtained with the automatic film scanner (1.92 volume percent) is about 2 percent larger than that obtained with the particle-size analyzer (1.88 volume percent). The differences between these measurements by the two methods are within the data spread usually encountered in quantitative metallography.

In comparison with the semiautomatic commercial particle-size analyzer, which itself is much faster than pencil-and-ruler methods, use of the overlay technique with an automatic film scanner reduced the time for analysis of dispersion microstructures by 90 to 95 percent. Such time saving is considerable since several thousand particles and several areas of the specimen should be counted in order to characterize the microstructure accurately.

Use of the overlay copy technique is not restricted to dispersion microstructures. It can be applied to any multiphase microstructure where phase boundaries or other microstructural features can be clearly defined. Examples of potential metallographic applications are measurements of the fraction recrystallized or the fraction transformed. An important advantage is that this technique allows preselection of the phases to be measured. Microstructural features, such as pores, unwanted phases, or false structures (artifacts) from improper etching or replication, can be eliminated or treated separately during the preparation of the overlay. It is believed that the overlay technique could be used advantageously with optical micrographs as well as with electron micrographs, and also with other measurement methods (such as the microdensitometer method of Allio and Randall (ref. 4)).

CONCLUSIONS

An overlay copy technique for preparing high-contrast film negatives of dispersion microstructures was applied to automatic quantitative metallography. The technique was

demonstrated with a replication electron micrograph of TD-Nickel, which was analyzed for particle-size distribution and volume-percent dispersoid with an automatic film scanner. The following conclusions were drawn:

1. The results were in good agreement with measurements made with the use of a commercial semiautomatic particle-size analyzer.

2. Microstructural parameters of dispersion alloys can be determined extremely rapidly compared with conventional methods of lineal analysis and particle sizing and counting.

3. Quantitative metallography of multiphase microstructures with an automatic film scanner has been made possible by the overlay technique. Preselection of phases or microstructural features to be measured can be made when the overlay is prepared.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, August 9, 1966,
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