222
10-6-69

1025

MASTER

RFP-1380

August 29, 1969

OK
T

A RELIABLE AND REPRODUCIBLE TECHNIQUE
FOR MACROSCOPIC ETCHING OF TYPES 304 STAINLESS STEELS

James F. Capes
James W. Snyder
Arthur E. Calabra

THE DOW CHEMICAL COMPANY
ROCKY FLATS DIVISION
P. O. BOX 888
GOLDEN, COLORADO 80401

U.S. ATOMIC ENERGY COMMISSION
CONTRACT AT(29-1)-1106

P2816

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of 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. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Atomic Energy Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

Printed in the United States of America

Available from

Clearinghouse for Federal Scientific and Technical Information

National Bureau of Standards, U. S. Department of Commerce

Springfield, Virginia 22151

Price: Printed Copy \$3.00; Microfiche \$0.65

August 29, 1969

RFP-1380
UC-25 METALS, CERAMICS
AND MATERIALS
TID-4500 - 54th Ed.

**A RELIABLE AND REPRODUCIBLE TECHNIQUE
FOR MACROSCOPIC ETCHING OF TYPES 304 STAINLESS STEELS**

James F. Capes
James W. Snyder
Arthur E. Calabra

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:
A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or
B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.
As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, in the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

**THE DOW CHEMICAL COMPANY
ROCKY FLATS DIVISION
P. O. BOX 888
GOLDEN, COLORADO 80401**

**Prepared under Contract AT(29-1)-1106
for the
Albuquerque Operations Office
U. S. Atomic Energy Commission**

CONTINUED ON THE FOLLOWING PAGE

Reg

A RELIABLE AND REPRODUCIBLE TECHNIQUE FOR MACROSCOPIC ETCHING OF TYPES 304 STAINLESS STEELS

James F. Capes, James W. Snyder and Arthur E. Calabra

Abstract. An extremely reliable procedure is described for macroscopic etching of 304 and 304-L type stainless steel forgings. The method is rapid and 100% reproducible. The surface of the forging to be examined is mechanically ground through a sequence of decreasing grit-size on silicon-carbide papers, including 600 soft grit. Prior to grinding, the entire forging is masked with a spray lacquer so that after grinding only the surface in question will be attacked by the etchant.

The surface to be macroscopically etched is immersed in or swabbed with an aqueous solution of cupric ammonium chloride and hydrochloric acid. The results show in fine detail the strain lines effected by the forging operation, and the forging is also ready for examination and photographing.

INTRODUCTION

Large forgings of 304 and 304-L stainless steel must be macro-etched to reveal strain lines (flow lines) induced by a forging operation. The flow-line configuration is the criterion upon which the quality of the forging is judged. Many of the macro-etching methods described in laboratory text books^{1, 2} require an acid, or a mixture of acids, heated to the boiling temperature. These etchants have proven to be cumbersome, time consuming, ineffective, potentially dangerous, and not reproducible.

The flow-line patterns resolved by some methods require an ink staining followed by a fine grinding to reveal the details for examination and photography. Therefore, other macro-etching procedures were thoroughly investigated. The authors believe that the procedure described in this report is the most efficient and reliable of the macro-etching techniques.

NOTE: Superscripts relate to references.

PROCEDURE

The surface preparation procedure is simple. First, the entire macro section is sprayed with lacquer.³ This will assure the technician that only the prepared surface in question will be attacked by the etchant. Experience has shown that a more efficient attack is effected by exposing only the area in question to the etching solution. The lacquer on the surface to be etched is removed by the subsequent grinding.

Grinding is performed using the normal sequence of decreasing grit-size on silicon-carbide papers; that is, 180, 240, 320, 400, and 600 grits. An additional fine grinding step, however, is used which makes the difference between a well-defined flow line and one of lesser quality. This is done using the 600 soft paper.⁴ This is a 600 silicon-carbide grit on a soft paper backing.

All grinding is done with copious amounts of cold water as a lubricant and coolant. Polishing beyond fine grinding is time consuming and will not increase the resolution of the forging flow lines.

When the surface preparation is completed, the surface in question is then immersed in the aqueous solution of cupric ammonium chloride and hydrochloric acid which is at room temperature. (See Table I.)

Etching time normally will take approximately 45 seconds. If the macro specimen is too large for an immersion, excellent results can be obtained by swabbing with cotton soaked in the etchant. The time for the swab technique will take 30 to 60 seconds longer.

The etchant as shown in Table I is a modification of Humfry's Reagent.^{1, 2} The hydrochloric acid is added only at the time of use. Fresh HCl appears to "liven" the etchant

(a)

(b)

FIGURE 1. Comparison photographs: (a) the remarkable results obtained using the procedure described, and (b) the results obtain using water — HCl (50:50), and ink stained. Observe the excellent resolution in the (a) photographs.

TABLE I. Macro-Etching Solution.

Stock solution	--	15 grams $\text{CuCl}_2 \cdot 2\text{NH}_4\text{Cl} \cdot 2\text{H}_2\text{O}$ (cupric ammonium chloride, reagent grade) 100 ml - distilled water
Working solution	--	100 ml stock solution 5 ml HCl
Temperature	--	Room temperature

resulting in the extremely fine, macroscopic, flow-line resolution. The working solution is used at room temperature.

After etching, the specimen is rinsed thoroughly with cold water and dried. The macroscopic structures are resolved to such fine detail that no additional preparation is required for examination and photography.

CONCLUSIONS

Figure 1 (a) shows the excellent forging detail obtained using the described procedure. Figure 1 (b) consists of comparison photographs of similar forgings but etched by using a boiling mixture of water and hydrochloric acid and an ink stain on the surface. The superior structural characteristics effected with the reported procedure are obvious.

REFERENCES

1. George L. Kehl, *Principles of Metallographic Laboratory Practice*, Third Edition, McGraw-Hill Book Company, Inc., New York, 1949.
2. R.S. Williams and V. O. Homerberg, *Principles of Metallography*, Fifth Edition, McGraw-Hill Book Company, Inc., New York, 1948.
3. Airosol Jet Clear Lacquer, Type II, Airosol Company, Inc., Neodesha, Kansas.
4. Microcut Polishing Paper, 600 soft grit, Buehler Ltd., 2120 Greenwood St., Evanston, Ill.