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Metallurgical Evaluation of Seamless Stainless Steel Cylinder

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards
National Measurement Laboratory
Center for Materials Science
Fracture and Deformation Division
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Final Report

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U.S. DEPARTMENT OF COMMERCE, Malcolm Baldrige, ***Secretary***
NATIONAL BUREAU OF STANDARDS, Ernest Ambler, ***Director***

Summary

A seamless cylinder fabricated from type 304 stainless steel was examined to assess the uniformity of properties and microstructure and to determine if the cylinder was in compliance with the DOT regulation governing these cylinders. The properties and microstructure did not show excessive variation throughout the cylinder. However, some evidence of carbide precipitation and sensitization were found in the cylinder. Recommendations are made to prevent possible sensitization in cylinders of this type.

Metallurgical Evaluation of Seamless Stainless Steel Cylinder

1. INTRODUCTION

1.1 Reference [1]

Office of Hazardous Materials Regulations, Department of Transportation, Washington, D.C. letter from A. J. Mallen to J. H. Smith, NBS. April 17, 1980, reference contract DOT-A-S-40034.

1.2 Background

Sections of a seamless cylinder from a lot of 12 cylinders manufactured by Autospin Inc., Carson, California were supplied by the Department of Transportation (DOT) to NBS for metallurgical and mechanical properties evaluation. The cylinders in the lot were 2.000-inches (5.080 cm) outside diameter, 16.250-inches (41.275 cm) long and had a minimum wall thickness of 0.183-inch (0.465 cm). The cylinders were made from seamless tubing of type 304 stainless steel (ASTM designation A511) and were made to comply with the DOT specification No. DOT-3A-500 (49 CFR part 178.36). The cylinders were manufactured by hot spinning the ends on sections of annealed, seamless, stainless steel tubing. The hot spinning was reported to have been done at a temperature of 1600°F (871°C) and was followed by water quenching.

The cylinders had a capacity of approximately 500 C.C. (30.5 cu. in.) and were designed to operate at a pressure of approximately 500 psi (3448 KPa) and were hydrostatically tested after manufacture to a maximum pressure of 833 psi (5744 KPa). At the test pressure, the calculated wall stress was 3926 psi (27070 KPa), based on 49 CFR part 178.36-10. The maximum allowable wall stress (49 CFR part 178.36-10) for a type DOT-3A cylinder is 24,000 psi (165493 KPa).

As specified in the DOT regulations (49 CFR part 178.36-11), all seamless cylinders must be uniformly and properly heat treated after fabrication and prior to conducting the required physical tests. The inspection report (1) shows that the lot of 12 completed cylinders, from which the test cylinder evaluated here was taken, was not heat treated after fabrication and prior to conducting the physical tests. However, the inspection report states that, because austenitic stainless steel, such as the type 304 used here, will not increase in strength during heat treatment, the final heat treatment requirement may not be necessary.

The cylinders in this lot did not strictly comply with the DOT regulations which require uniform heat treatment after fabrication. Therefore, it was decided to assess the significance of this lack of final heat treatment by determining, 1) the uniformity of the metal properties and 2) if the metallurgical structure is essentially the same as that which would result from heat treating the completed cylinders (1).

2. EXPERIMENTAL WORK AND RESULTS

2.1 Evaluation of the Uniformity of Properties

To evaluate the uniformity of the completed cylinder, the cylinder was sectioned longitudinally and hardness measurements were made on the neck, sidewall, and bottom of the cylinder. The hardness results are shown in Figure 2. These results show that the hardness is quite uniform throughout the cylinder and is typical of that of annealed stainless steel of type 304. The hardness of annealed 304 stainless is specified to be in the range of 75 to 90 on the Rockwell B scale (2) variation from a hardness of 88 R_B at the cylinder neck to a hardness of 84 R_B along the cylinder sidewall were observed. This range of hardness is within the normally expected variation for a uniform wrought stainless steel product and indicates that the cylinder was essentially annealed with only a very minimum amount of cold work during fabrication of the cylinder. The bottom of the cylinder, which was hot spun and water quenched, exhibited hardness levels of 78 R_B to 85 R_B. These hardness levels are slightly below the hardness levels found in the sidewall and neck of the cylinder. This indicates that some slight softening due to annealing and possibly grain coarsening occurred during the hot spinning of the cylinder bottom. For comparison, a sample was cut from the sidewall of the cylinder, fully annealed for 1 hour at 2000°F (1093°C) and water quenched. This fully annealed sample had hardness measured in the range 73 R_B to 77 R_B. This indicates that the bottom of the cylinder, which had a hardness of 78 R_B to 82 R_B, was essentially fully annealed.

Overall, the hardness measurements, which ranged from 78 R_B to 88 R_B throughout the completed cylinder, indicate that the properties of the metal in the cylinder are only slightly more variable than would be expected if the cylinders were fully heat treated by annealing after completion of the fabrication of the cylinder. As indicated by the fully annealed test sample, further annealing might lower the hardness even further to the 73 R_B to 77 R_B range due to grain coarsening.

2.2 Evaluation of the microstructure

Optical photomicrographs were taken at several locations throughout the cylinder to assess the uniformity of the microstructure. The locations on the cylinder at which photomicrographs were taken are shown in Figure 3.

The microstructure shows a slight amount of cold work at the neck of the cylinder as indicated by slightly distorted and elongated grains (Figure 4 and 5, Photomicrographs A and B). The remainder of the cylinder has a microstructure that is primarily equiaxed grains typical of annealed stainless steel (Fig. 6, 7, 8, 9, 10, 11, 12, 13 corresponding to locations C, D, E, F, G, H, I, J of Fig. 3). The microstructure at the bottom of the cylinder (Fig. 12 and 13), which was formed by hot spinning, shows somewhat larger equiaxed grains than the rest of the cylinder which indicates that some grain coarsening occurred during the hot spinning.

2.3 Evaluation of susceptibility to carbide precipitation and investigation

Type 304 stainless steel is known to be susceptible to a phenomena known as sensitization which significantly lowers its resistance to intergranular corrosion (3). Sensitization occurs due to the precipitation of carbides at the grain boundaries when the steel is held at temperatures between 800°F (427°C) and 1600°F (871°C) or when it is cooled slowly through this temperature range. The presence of sensitization will not be detected by changes in properties, such as hardness, but can be detected from examination of the microstructure by etching with oxalic acid (4) to highlight the presence of carbides in the grain boundaries.

Photomicrographs taken at the bottom of the cylinder (Fig. 12, location I) showed no carbide precipitation and sensitization. This indicates that the bottom of the cylinder which was hot spun, was heated sufficiently (above approximately 1600°F/871°C) during hot spinning and was properly water quenched so that the carbon remained in solution in the grains and did not precipitate and cause sensitization.

Photomicrographs taken along the sidewall of the cylinder but near the bottom (Fig. 8, 9, 10 at locations E, F, G) did show evidence of carbide precipitation at the grain boundaries. This indicates that although the bottom of the cylinder was heated sufficiently to avoid sensitization, the sides of the cylinder a short distance back from the bottom were heated by conduction into the critical temperature range to cause sensitization.

To assess the susceptibility to sensitization, sections of the cylinder were reheat treated to simulate the range of heat treatments that the cylinders might receive. The following heat treatments were performed:

Temperature	Cooling	Objectives
A. 2000°F/1093°C	Water Quench	1. To demonstrate full and proper annealing 2. To show absence of sensitization
B. 1200°F/649°C	Air Cooled	1. To demonstrate worst case of sensitization

The photomicrographs from these tests are shown in Figure 14 for heat treatment A and in Figure 15 for heat treatment B. As expected, these tests show that if the cylinder is fully and properly annealed by heating to 200°F (1093°C) and water quenched, all evidence of sensitization is absent. However, if heated within the critical temperature range of 800°F (427°C) to 1600°F (871°C) and slow cooled, severe sensitization will occur.

3. DISCUSSION

3.1 Uniformity of Properties

The hardness measurements taken throughout the cylinder that was supplied, indicated that the properties of the cylinder were generally uniform and were within the range expected for annealed 304 stainless steel (2). The hardness at the bottom of the cylinder, which had been hot spun and water quenched, was slightly lower than the hardness of the rest of the cylinder due to grain coarsening. The specimen that was taken from the cylinder and fully annealed had a hardness in the range of 73 R_B to 77 R_B, which indicates that the entire cylinder may have been slightly cold worked during fabrication (hardness ranged from 78 R_B to 88 R_B). However, any cold working was not sufficient to be detected metallographically. Therefore, a final heat treatment of the cylinder for purposes of annealing would not result in any significant change in the properties of the cylinder or any substantial improvement in the uniformity of the properties.

3.2 Uniformity of the Metallurgical Structure

The microstructure was essentially equiaxed grains throughout the cylinder representative a fully annealed microstructure. A very limited amount of cold working was evident at the neck of the cylinder. Slightly enlarged, equiaxed grains were observed in the hot spun bottom of the cylinder. However, this amount of grain coarsening is not expected to result in significant degradation of the strength or other mechanical properties of the cylinder. For comparison, the sample of the cylinder that was reheat treated by annealing at 2000°F (1093°C) and quenched in water to simulate fully annealing the cylinder shows grain coarsening similar to that observed in the bottom of the cylinder (Fig. 14). This annealing resulting only slightly lowering the hardness to a range of 73 R_B to 77 R_B.

3.3 Evaluation of the need for Uniform Heat Treatment

As discussed above, the properties and microstructure of the cylinder are nearly uniform throughout the completed cylinder. Additional heat treatment by annealing would not substantially change the properties or microstructure. At most any annealing would only slightly soften the cylinder and cause some coarsening of the grain structure.

However, annealing followed by water quenching would remove any carbide precipitation at the grain boundaries which leads to sensitization and can cause a significant decrease in the corrosion resistance of the cylinder. Some sensitization was observed in the sidewall of this cylinder just above the hot spun bottom. The carbide precipitation and sensitization is a reversible process and is removed by annealing of the stainless steel at temperature above 1600°F (871°C) followed by rapid cooling (water quenching). As shown by Figure 14, all traces of sensitization were removed by a proper anneal of a section of the cylinder. Therefore, if the fabrication of the cylidner causes it to be heated within the critical temperature range of 800°F (427°C) to 1600°F (871°C) where sensitization can occur, it is recommended that a final annealing of the complete cylinder be performed.

3.4 Assessment of the Adequacy of the Cylinder Design and Material

The present cylinders are designed to operate at a pressure of 500 psi (3448 KPa) and to be proof tested at a pressure of 833 psi (5744 KPa). At the proof test pressure of 833 psi (5744 KPa) the calculated wall stress is 3926 psi (27,070 KPa). The measured minimum hardness on the cylinder was 78 R_B which corresponds to a yield strength of approximately 30,000 psi (206,867 KPa) and a tensile strength of 75,000 psi (517167 KPa). This indicates that even at the proof test pressure, the wall stress is less than 1/7 of the minimum yield strength of the cylinder, so that there is a very large margin of safety against failure by burst. In addition, the elongation to fracture was reported to be 44% (1), which indicates very high ductility and resistance to fracture.

4. CONCLUSIONS

4.1 The stainless steel cylinders as fabricated are adequate for their intended use. The microstructure and properties do not show excessive variation throughout the cylinder. A wide margin of safety against failure by bursting exists because the wall stress, even at the proof test pressure, is less than 1/7 of the yield strength of the material in the cylinder.

4.2 The cylinder showed some evidence of sensitization in a limited region which can result in an increase in the susceptibility to intergranular corrosion. This sensitization cannot be detected by hardness measurements or during proof testing. The sensitization, when it occurs can be removed by fully annealing the cylinder after fabrication.

5. RECOMMENDATIONS

5.1 The DOT regulations as presently written (49 CFR part 178.36) specify that uniform and proper heat treatment should be done after the cylinder is fabricated and prior to conducting the physical tests. It would be more correct if the regulations were revised to distinguish between cylinders requiring heat treatment for the purpose of increasing the strength of the metal, such as carbon steels, and cylinders that are heat treated by annealing, such as austenitic stainless steel cylinders, for the purpose of increasing the ductility and corrosion resistance of the cylinder. For cylinders that are strengthened by heat treatment, a uniform and proper heat treatment should be mandatory. Cylinders made from metals that are not strengthened by heat treatment, should only be required to meet satisfactory strength and ductility requirements.

5.2 For cylinders made from austenitic stainless steel, the DOT regulations should be revised to minimize or eliminate the possibility of sensitization during fabrication. This may be done by restricting the fabrication of these cylinders to stabilized grades of austenitic stainless steel such as types 321 or 347, or to extra low carbon grades such as types 304L or 316L. If type 304 or 316 stainless steel is permitted for these cylinders, a proper heat treatment to a temperature of at least 1600°F (871°C) followed by water quenching should be required to remove any trace of sensitization after fabrication.

REFERENCES

1. Memorandum, April 17, 1980 from A. J. Mallen, Office of Hazardous Materials Requlations, Dept. of Transportation to J. H. Smith, National Bureau of Standards.

2. "Aerospace Structural Metals Handbook," Mechanical Properties Data Center, Belfour Stelun Inc., Traverse City Michigan, 1975 ed., Section 1303 pg. 1-29.

3. "Source Book on Stainless Steels," American Society for Metals, Metals Park, Ohio, 1976, pg. 162.

4. "Standard Recommended Practices for Detecting Susceptibility to Intergranular Attack in Stainless Steel" ASTM Standard A-262.

Fig. 1. Sections of Cylinder As Received.

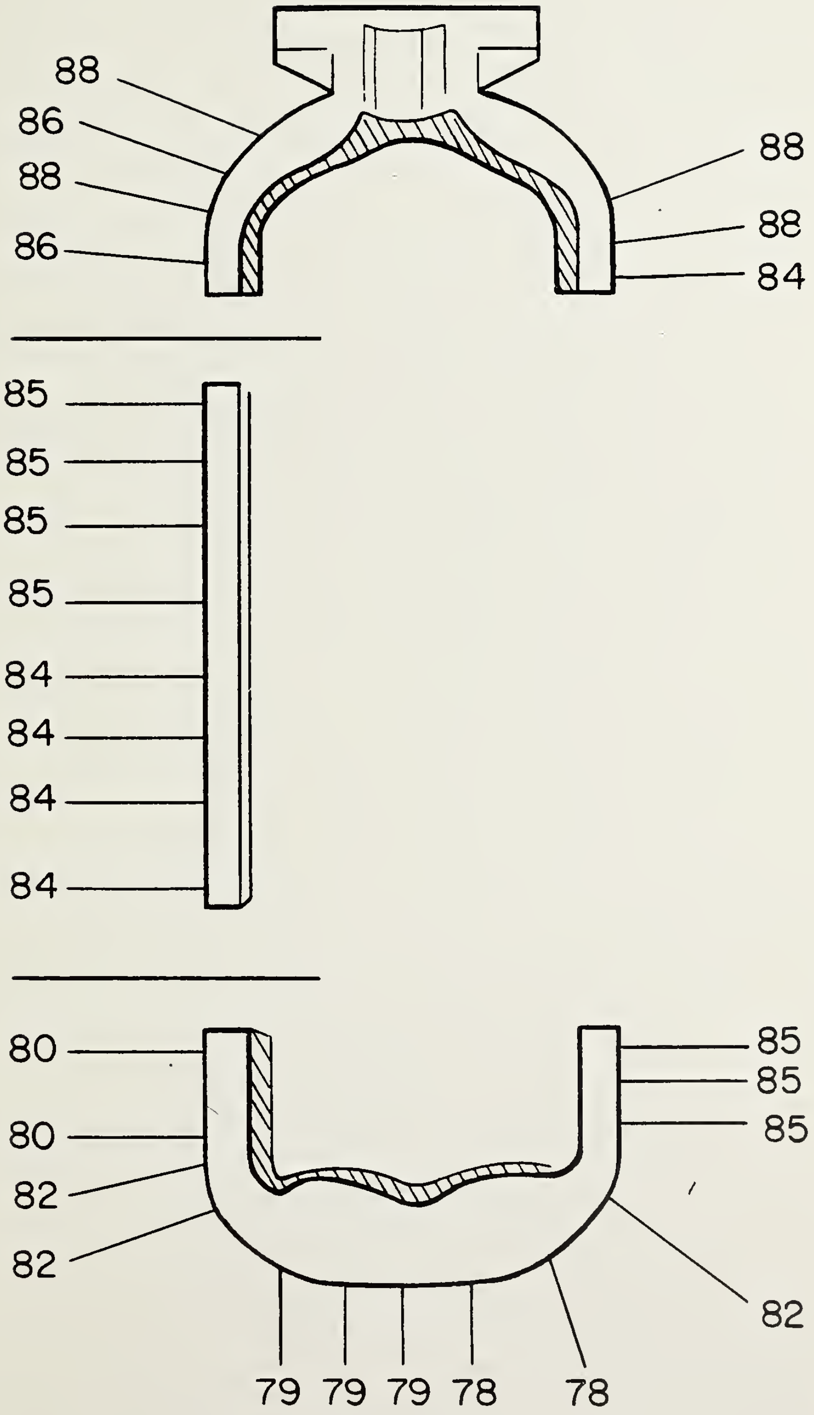

FIG. 2 HARDNESS MEASUREMENTS (ROCKWELL B SCALE)

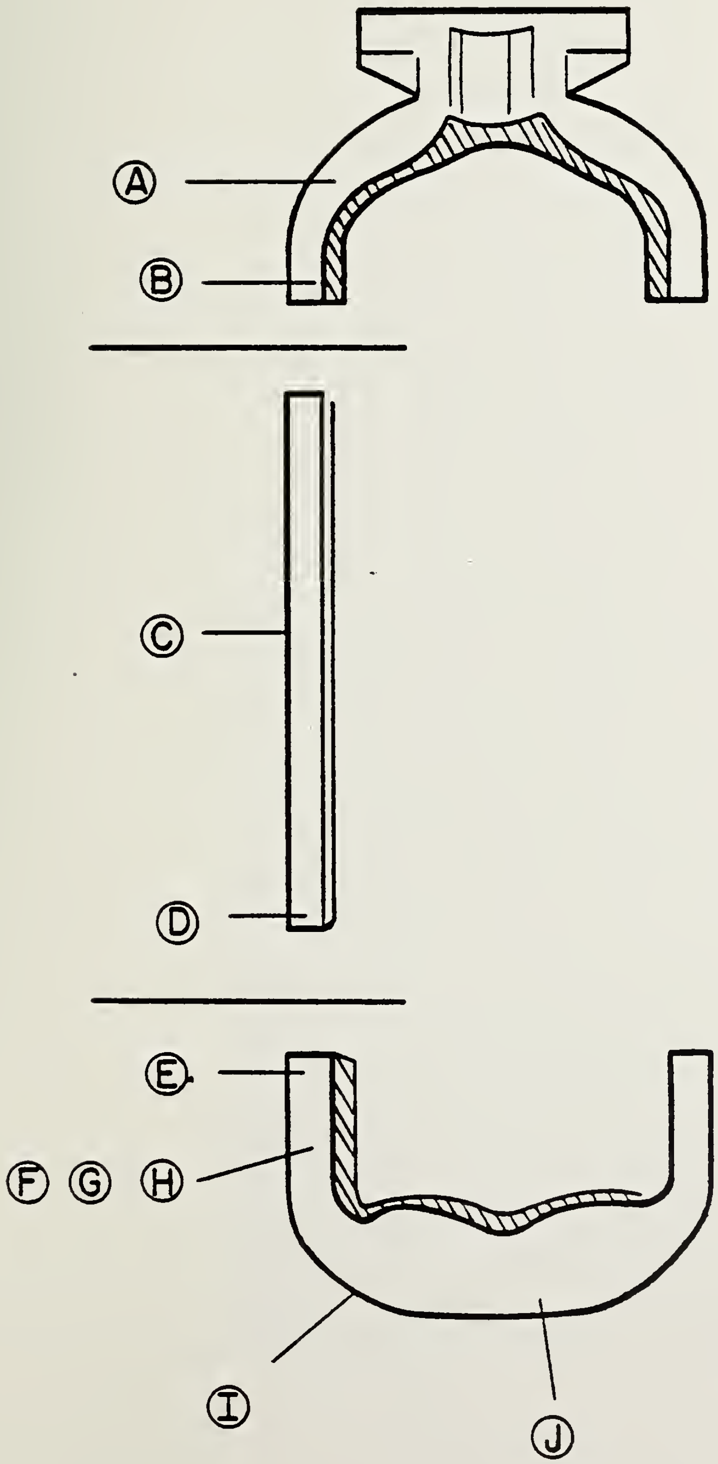

FIG. 3 LOCATION OF PHOTOMICROGRAPHS

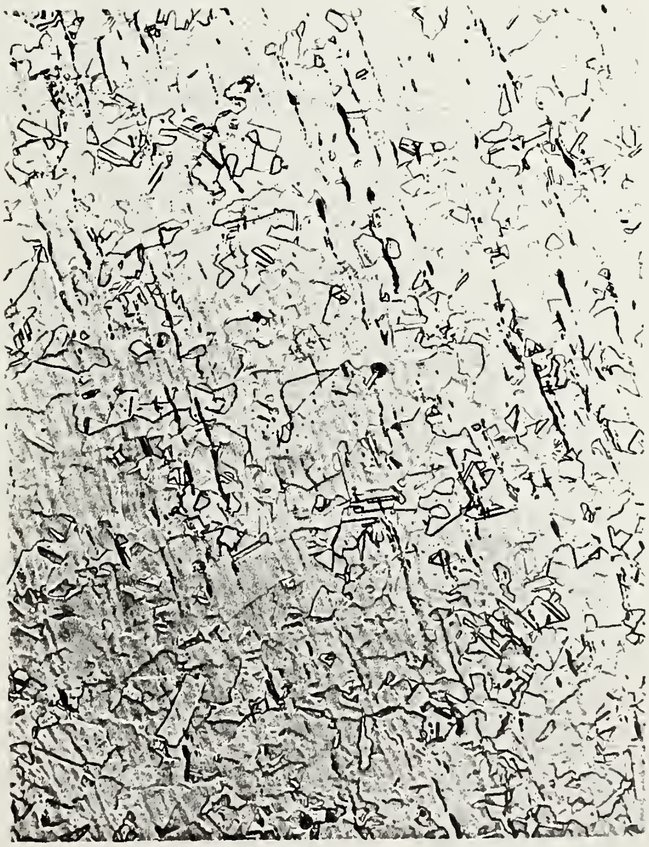

Fig. 4. Photomicrograph at Location A showing slightly deformed grains. Mag. 100X

Fig. 5. Photomicrograph at Location B showing slightly deformed grains. Mag. 100X

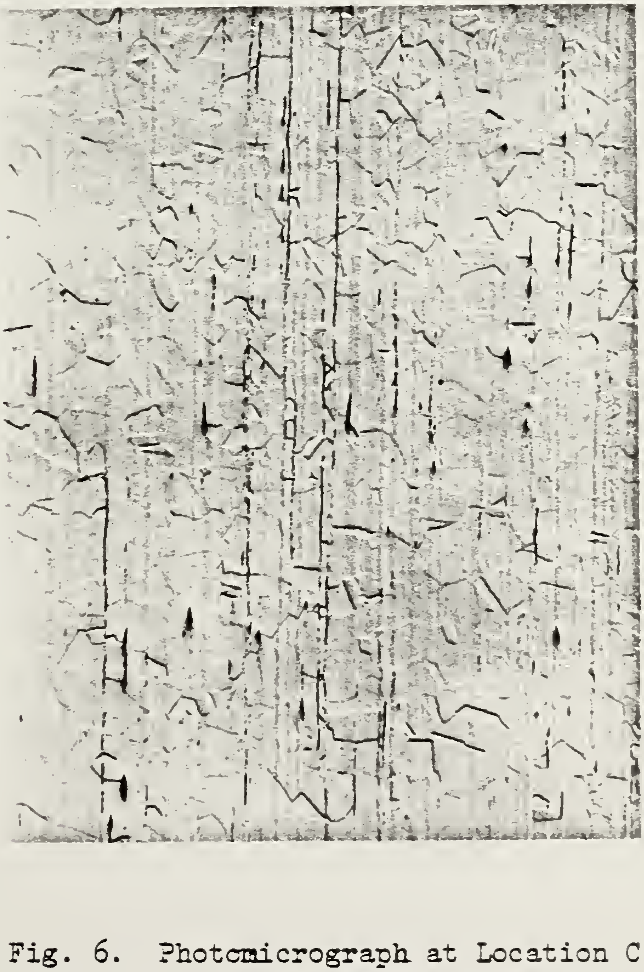

Fig. 6. Photomicrograph at Location C showing equiaxed grain structure. Mag. 100X

Fig. 7. Photomicrograph at Location D showing typical ferrite stringer. Mag. 500X

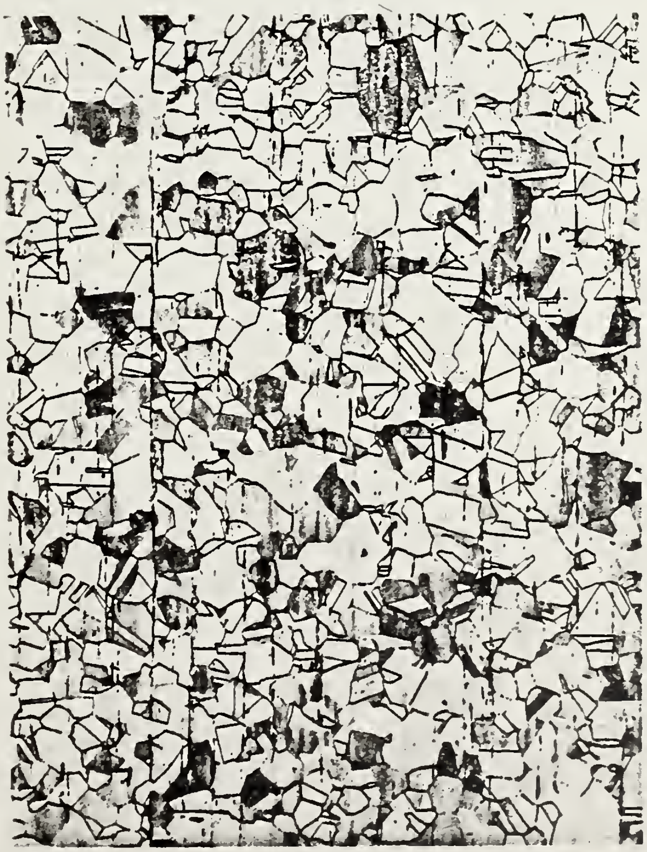

Fig. 8. Photomicrograph at Location E
showing carbide precipitation at
grain boundaries. Mag. 100X

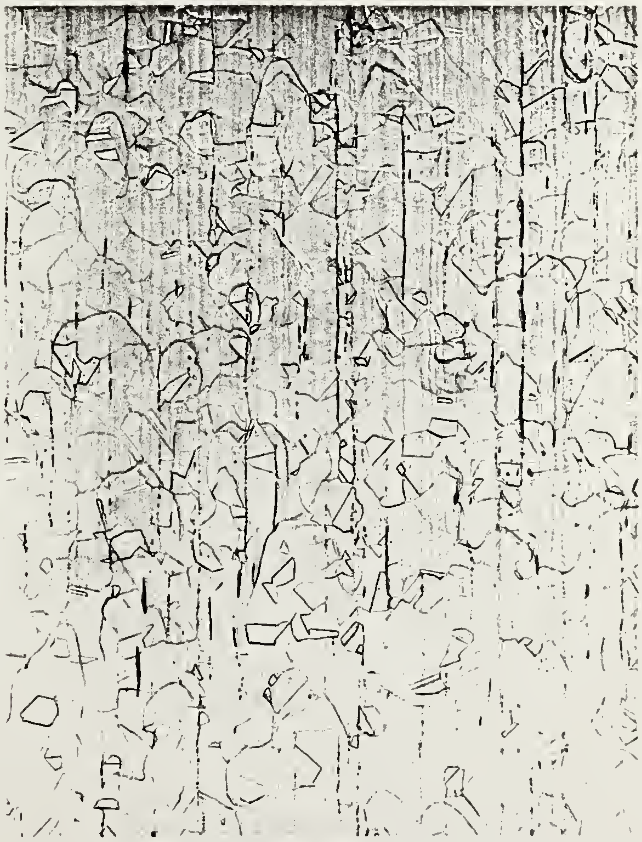

Fig. 9. Photomicrograph at Location F showing equiaxed grain structure. Mag. 100X

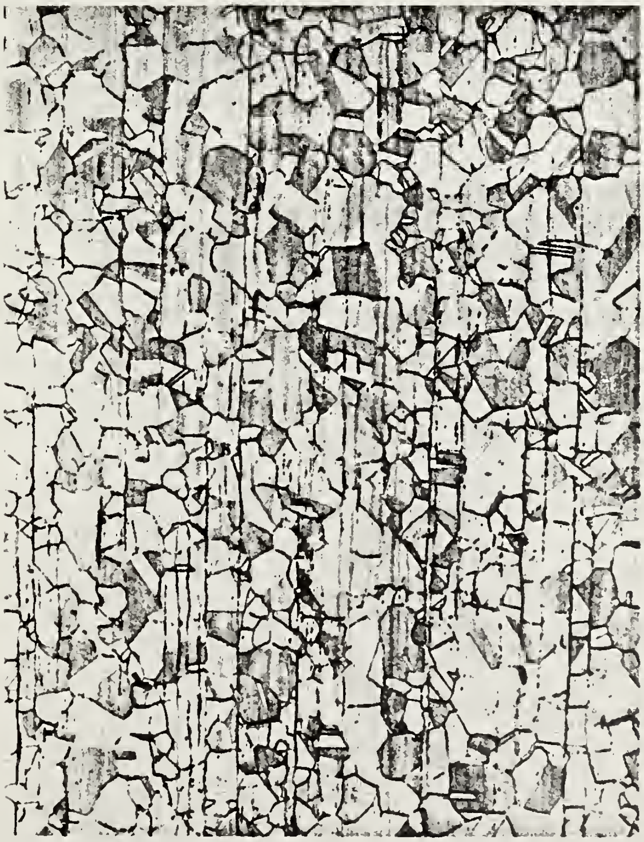

Fig. 10. Photomicrograph at Location G
showing carbide precipitation.
Mag. 100X

Fig. 11. Photomicrograph at Location H showing equiaxed grains. Mag. 200X

Fig. 12. Photomicrograph Location I showing no carbide precipitation. Mag. 100X

Fig. 13. Photomicrograph Location J showing enlarged equiaxed grains. Mag. 100X

Fig. 14. Photomicrograph of reannealed specimen 2000°F/water quenched. Mag. 100X

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10. SUPPLEMENTARY NOTES

☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.

11. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)*

A seamless cylinder fabricated from type 304 stainless steel was examined to assess the uniformity of properties and microstructure and to determine if the cylinder was in compliance with the DOT regulation governing these cylinders. The properties and microstructure did not show excessive variation throughout the cylinder. However, some evidence of carbide precipitation and sensitization were found in the cylinder. Recommendations are made to prevent possible sensitization in cylinders of this type.

12. KEY WORDS *(Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)*

Metallurgical evaluation; Pressure vessels; Stainless steel; Stainless steel, austenite.

13. AVAILABILITY

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