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# Shot Peening to Prevent the Corrosion Cracking of Austenitic Stainless Steels

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Rockwell International and Metal Improvement Company have conducted a joint test program<sup>1</sup> to develop shot peening as a technique for preventing corrosion cracking in austenitic stainless steels. Initial laboratory-scale tests demonstrated the feasibility of preventing stress corrosion cracking by shot peening to impose compressive stresses on the surface of the work pieces. Conventional U-bend test specimens, when peened, survived 1000 h tests in the boiling 42 pct magnesium chloride stress corrosion test. Unpeened reference specimens commonly fractured within one or two hours in this test. Component tests were conducted to demonstrate the practicality of the peening process for sizes and shapes that typify components in a reactor piping system. Pipe sections and cold worked, hexagonal tubes were tested. In all components, unpeened sections developed stress corrosion cracks within a few hours in the magnesium chloride test; in contrast, the shot peened surfaces survived hundreds of hours. It was discovered at Rockwell International that intergranular corrosion can be prevented in austenitic stainless steels by severe shot peening prior to exposure to sensitizing temperatures. \*/For this purpose, the surfaces must be severely

\*U.S. Patent No. 3,844,846. Metal Improvement Company, Licensee.

cold worked by the shot peening to break up surface grains and grain boundaries. Two nondestructive testing techniques show promise as methods for measuring the stresses or cold work imparted on the surface of the work piece by peening. In one method, eddy currents are used to measure differences in electrical properties induced by cold working of metals. Another is a magnetic technique which measures the changes in magnetic properties due to the transformation of austenite to ferrite by cold working. enel

# I. INTRODUCTION AND BACKGROUND

One of the few shortcomings of the 18-8 type of austenitic stainless steels is their susceptibility to stress corrosion cracking and intergranular corrosion. It has long been known that a proper cold working surface treat-

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ment could be a preventative control measure for both of these modes of corrosion. Stress corrosion cracking (SCC) can occur only in the presence of a tensile stress; therefore, it can be prevented by imposing a compressive stress on the surface exposed to the corrosive environment. Compressive stresses can be induced by such processes as shot peening, cold rolling, swaging or tumbling. Nevertheless, the application of this principle for controlling SCC is not widely practiced in industry, at

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woodif JEMATERIALS FOR ENERGY SYSTEMS least not for the stainless steels. With respect to intergranular corrosion (IGC), E. C. Bain, et al1 demonstrated some 40 years ago that cold working of austenitic stainless steels by cold rolling greatly reduced their susceptibility to intergranular attack if subsequently exposed to sensitizing temperatures. However, because the entire bulk of the alloy had to be treated, excessive work hardening occurred. Thus as late as 1967 it was still being published that cold working for this purpose was not a recommended or practical procedure.<sup>2</sup> The preferred methods of corrosion control have been and continue to be 1) heat treatments to relieve residual stresses and to minimize the risk of SCC, 2) solution annealing to eliminate chromium-depleted zones and remove grain boundary carbides, and 3) stringent control of the environments to minimize the contamination that can promote either or both of these corrosion modes.

This report addresses the application of one specific cold working procedure, controlled shot peening, as a practical method of preventing both SCC and IGC. Firstly, shot peening imposes a compressive stress on the surface that can negate the tensile stresses, residual or applied, that are required for SCC. Secondly, shot peening uniformly cold works the surface of the work piece; therefore, it can effectively reduce susceptibility to IGC. Thirdly, the controlled shot-peening process is adaptable to most wrought or cast products or fabricated components, regardless of size and shape.

Shot peening is the controlled impingement of a stream of high velocity shot on metal surfaces. As each individual shot bombards the work piece, it forms a de-



Fig. 1-Effects of shot-peening process variables on residual stress.



Fig. 2—Stress distribution in a shot-peened beam: (a) distribution of stress in a shot-peened beam with no external load, (b) resultant distribution of stress in a shot-peened beam with external load applied; solid line is the resultant.

pression by plastic deformation of the surface grains, thereby stretching the surface radially to cause tensile stresses. As this shot bounces off, the tensile stresses are relieved and the resultant surface stresses at equilibrium become compressive. When saturated by a multitude of shot impacts, the entire surface of the peened area is cold worked by plastic deformation and is compressively stressed.

Figure 1 is a series of curves that illustrate the stress profiles of carbon and stainless steel when peened under various process parameters. For Type 304 stainless steel, the compressive stress level was determined to be about 70,000 psi (483 Pa) at the surface and the compressive layer extended to a depth of about 15 mils (0.038 cm). The curves for carbon steel indicate the effects of shot size and peening pressure on the stress profiles.

Figure 2 depicts the stress pattern of a shot-peened beam in no-load and applied load conditions. Note that the peened surface is in compression in the absence of an external load. When a bending load is applied, the surface will remain in compression until the applied tensile stress exceeds the residual compressive stress induced by shot peening.

Figure 3 shows the microstructure of peened and unpeened Type 304 stainless steel. Note the severely cold worked microstructure that is characteristic of shotpeened surfaces as utilized in this process. As discussed later, such severe shot peening is essential to prevent in-



Fig. 3—Photomicrographs of peened and unpeened Type 304 stainless steel plate surfaces; (a) shot peened with 0.028 in. (0.071 cm) diam shot to Almen Intensity 0.010A, (b) as received (unpeened).

tergranular corrosion as it 1) provides a multitude of slip planes, twin boundaries and dislocations as nucleation sites for carbide precipitation within grains rather than grain boundaries and 2) breaks up the continuous grain boundaries typical of annealed microstructures that lead to intergranular attack if sensitized.

The tests and evaluations reported herein were con-

ducted to confirm the feasibility of the shot-peening principles for controlling both modes of corrosion in the austenitic stainless steels and to assess its practicability for industrial components and systems. Included are the results of 1) scoping tests intended to assess the effects of various peening process variables, particularly as related to service conditions of Liquid Metal Fast Breeder Reactor (LMFBR) systems, 2) scaleup tests in which representative welded and unwelded pipe sections were evaluated, 3) intergranular corrosion tests of peened and unpeened specimens after being exposed to sensitizing temperatures, and 4) nondestructive tests to assess and control the effectiveness of the shot-peening process. In addition, the known problem areas and development needs are discussed.

### **II. MATERIALS AND PROCEDURES**

The materials covered in this study were the austenitic stainless steel Types 304, 316, 321, and 347. Conventional U-bend specimens used in the SCC scoping tests were of Type 304 stainless steel strip, 0.090 in. (0.23 cm) or 0.125 in. (0.32 cm) thickness. The coupons used for intergranular corrosion tests were of Type 304 stainless steel as 1/4 in. (0.64 cm) thick plate. The scaleup welded pipe specimens were of Type 321 or 347 stainless steel while the  $41/_2$  in. (11.4 cm) hexagonal tube was Type 316 stainless steel. The 8 ft (244 cm) length of 6 in. (15.2 cm) diam pipe was' Type 304 stainless steel. Except for the Type 316 stainless steel hexagonal tube which was 20 pct cold worked, all material was in the solution annealed condition.

Most of the peening was done by Metal Improvement Company using commercial peening equipment and procedures. Some of the parametric test specimens were peened at Rockwell International using a converted abrasive grit blaster. All test specimens were peened manually, with the exception of the 8 ft (244 cm) long, pipe section which was peened by a fully automated process on both outside and inside diameters.

The conventional peening material was cast steel shot, Grade 230 [nominally 0.023 in. (0.58 mm) diam] or Grade 280 [0.028 in. (0.71 mm) diam] conforming to MIL-S-851. The peening of the pipe section was with a smaller size, Grade 170 [0.017 in. (0.43 mm) diam], of hardness 58 to 63  $R_c$ . For some experimental peening studies, specimens were peened with glass beads or by Metal Improvement's proprietary process using ceramic beads.

The effectiveness of the peening processes was evaluated by conventional tests for stress corrosion cracking or intergranular corrosion. For SCC, the specimens were immersed in boiling 42 pct magnesium chloride. The evaluation criteria were the time to initiate stress corrosion cracks and/or time to failure of the specimen. In this test, cracks commonly initiated in about 1/2 h in unpeened U-bend specimens (used for control purposes), with full failure generally occurring within 2 h. For IGC, the specimens were immersed in a 10 pct HNO<sub>3</sub>-3 pct HF solution at 70 °C and examined metallographically for intergranular attack.

# **III. TEST RESULTS**

#### A. Scoping Tests

The scoping phase of the study was intended to establish the feasibility of the shot-peening process to prevent the stress corrosion cracking and intergranular corrosion of austenitic stainless steels. The factors evaluated included such process variables as peening intensity (i.e., pressure, time, distance, etc.), shot material and size, and coverage; the effects of elevated temperatures on the decay of the compressive stresses imposed by shot peening and the concomitant effects on resistance to SCC; the effects of exposures at sensitizing temperature with respect to intergranular attack; and the effectiveness of shot peening in preventing SCC due to residual stresses in weldments. A conventional U-bend specimen design was used in these tests. Figure 4 shows peened and unpeened surfaces of the U-bends after the SCC test in MgCl<sub>2</sub>. Note the stress corrosion cracks in the unpeened surface and the absence of cracks in the peened specimen.

Table I. Shot Peening—Scoping Tests Summary

Parameter	Comments		
Peening Intensity	Unpeened U-bend specimens stress corrosion cracked in 1 to 2 h in boiling MgCl <sub>2</sub> test; specimens peened over a wide range of peen- ing intensities survived 1000 h without crack- ing.		
Shot Material	Peening is effective with steel, glass, or ceramic beads.		
Stress Decay	Peening is effective after thermal soaking at LMFBR operating temperatures [1050 °F (566 °C)].		
Sensitization (Stress Corrosion)	Peening is effective on sensitized material.		
Sensitization (Inter- granular Corrosion)	Peening may prevent intergranular corrosion.		
Weld Stresses	Stresses induced by welding are sufficient to cause stress corrosion cracking; peened welds did not crack.		
Peening Defects	Process control required to assure 100 pct peening coverage.		

The results of these scoping tests, summarized in Table I, are discussed in the following paragraphs.

#### **1. PEENING PARAMETERS**

Two series of U-bend specimens were prepared by peening to three Almen Intensity levels to determine if resistance to stress corrosion was affected by variations





Fig. 4—Type 304 stainless steel U-bend specimens after stress corrosion tests in boiling 42 pct magnesium chloride: (a) unpeened, (b) shot peened.

in the shot-peening process. Each series consisted of nine Type 304 stainless steel specimens, three each being peened to Almen Intensities\* of 0.004A, 0.007A, and

\*Almen Intensity is an industry standard for measuring, specifying, and controlling the shot peening process. Refer to the SAE Manual on Shot Peening, AMS 2430, or MIL-S-13165.

0.011A. These specimens, plus unpeened control specimens, were tested by immersing in boiling 42 pct MgCl<sub>2</sub>. All 18 shot-peened specimens completed more than 1000 h in test without cracking. In comparison, all unpeened control specimens initiated stress corrosion cracks within ~1 h. Table II summarizes the peening process conditions and results for one of the parametric series. These tests demonstrated that shot peening at Almen Intensity levels as low as 0.004A will effectively combat SCC in austenitic stainless steels for apparently unlimited periods of time under very corrosive conditions. Similarly, the results indicated that peening at intensity levels as high as 0.011A does not "overpeen," i.e., initiate surface or subsurface microcracks or other defects, or otherwise reduce the effectiveness of the peeninginduced compressive stresses. The primary requirement for peening within this range of intensities is the assurance of complete coverage of the surface. As shown in these tests, peening at "200 pct coverage" is adequate.

### 2. SURFACE COVERAGE TESTS

U-bend specimens were prepared with intentionally unpeened areas to determine the degrading effect of

Table II. Results of Parametric Tests (Type 304 Stainless Steel U-Bend Specimens)

Peening Parameters*			SCC Test	
Specimen Number	Almen Intensity	Air Pressure (psig)	Time to Initiate Cracks (h)	
4-40	0.004A	40	>1005, NF†	
5-40	0.004A	40	>1005, NF	
6-40	0.004A	.40	>1005, NF	
4-70	0.008A	70	>1005, NF	
5-70	0.008A	70	>1005, NF	
6-70	0.008A	70	>1005, NF	
4-100	0.011A	100-110	>1005, NF	
5-100	0.011A	100-110	>1005, NF	
6-100	0.011A	100-110	>1005, NF	
Control Unper 42 <sup>1</sup> / <sub>2</sub> , 48 <sup>1</sup> / <sub>2</sub> , 12	ened. Tested at 0 h 1, 310, and 700 h		-1 h	

\*U-Bend specimens peened with 230 grade steel shot.

†NF-No failure, test terminated.

such defects on resistance to SCC. Incomplete coverage was simulated by applying a tough plastic tape to the stressed surface of the specimen prior to peening, and removing it before stress corrosion testing to expose unpeened areas. Three types of simulated defects were produced: 1) several small unpeened areas [about  $\frac{1}{8}$  in. (0.318 cm) by  $\frac{3}{16}$  in. (0.48 cm)] on the stressed surface, 2) a narrow unpeened strip  $[\frac{3}{16}$  in. (0.48 cm) wide] that was parallel to the direction of the applied tensile stresses, and 3) an unpeened strip that was transverse to the applied stresses. Two series of such specimens were subjected to the boiling MgCl<sub>2</sub> test. Five of the six specimens developed stress corrosion cracks that originated in the simulated peening defects. For small defects, the adjacent peened surface acted as a crack stopper; for others, the cracks continued to propagate through the peened surface. One of the specimens, type 1) above, survived 99 h in the SCC test without cracking. These simulated defect tests demonstrated that SCC can take place in unpeened areas where tensile stresses are present and indicate the need for complete peening coverage. While the adjacent compressively stressed peened surfaces resist propagation of cracks, they will not prevent it if the tensile stresses are sufficiently high.

### 3. ALTERNATE PEENING MATERIALS

In addition to the conventional cast steel shot, tests were conducted to evaluate the effects of peening with glass beads and with ceramic beads. In normal peening practice, steel shot leaves an iron residue on the work piece; the use of a non-steel peening media could possibly eliminate this iron contamination concern.

Three U-bend specimens were peened with glass beads to Almen Intensities of 0.004A, 0.006A, and 0.008A, and tested in the MgCl<sub>2</sub> test. The specimen peened to 0.008A completed 1007 h without cracking; the other two specimens failed prematurely (>200 h) due to stress corrosion cracking at the unpeened bolt holes in the legs of the specimens, not in the highly stressed loop of the U-bend. These tests indicated that peening with glass beads only provides adequate compressive stresses to provide protection against SCC. The premature failure of two of the three specimens was unrelated to the use of glass beads since failure occurred in an unpeened area.

Experiments were also conducted to determine the feasibility of ceramic beads as the peening media. A batch of 390 grade ceramic beads of a proprietary composition (a mixture of alumina and titania) was procured, conditioned to attain a smooth, spherical surface, and used for experimental peening studies. It was determined that higher levels of Almen Intensity could be attained with these ceramic beads as compared to

those of steel shot. To illustrate, for the peening conditions to attain Almen Intensities of 0.006A, 0.008A, 0.011A and 0.013A with steel shot, the resultant Almen Intensities with the ceramic beads were 0.009A, 0.015A, 0.019A and 0.022A, respectively. Subsequent corrosion tests indicated that ceramic peened surfaces were effective in preventing both SCC and IGC.

#### 4. STRESS DECAY

One possible deficiency of shot peening is the relief of the peening-induced compressive stresses by exposures to elevated temperatures. Heating to the 1000 to 1050 °F (538 to 570 °C) temperature range can be expected to reduce the residual stress level by about 35 pct in ~1 h with little additional stress relief at longer exposures.<sup>5</sup> To determine how stress relief of this magnitude would affect SCC resistance, peened and unpeened U-bend specimens were stressed, heated, and tested in MgCl<sub>2</sub>.

In the first test, the specimens were heated to  $1000 \,^{\circ}\text{F}$  (538 °C) for 16 h prior to testing. In the second test, the specimens were heated to  $1050 \,^{\circ}\text{F}$  (570 °C) which approximates the maximum LMFBR operating temperature for a period of 144 h. Although the expected stress relief at these temperatures occurs within ~1 h, the longer times are more representative of startup and initial full power operation.

The results, summarized in Table III, show that the SCC resistance of peened surfaces is retained, even though the level of the compressive stresses is reduced by exposures to 1000 to 1050 °F (538 to 570 °C) temperature. None of the peened specimens failed in the SCC test, confirming the retention of a compressively stressed surface after heating. The thermal treatment extended the time-to-failure of the unpeened specimens to 7 to 10 h, compared to ~1 h for nonheat-treated specimens, in-

	Table III. Effect of Stress Relief on
Stress	<b>Corrosion Resistance of Peened Type 304</b>
	Stainless Steel U-Bend Specimens

Stress Relief Temperature	Time (h)	Specimen	Tensile Stress Corrosion Cracking
1000 °F (538 °C)	16	Unpeened-1 Unpeened-2 Peened*	3 h 7 103 NF†
1050 °F (570 °C)	144	Unpeened Peened*	10 h 202 NF

\*Peened to 0.008 Almen by Metal Improvement Co. †NF—No failure, test terminated.



Fig. 5—Intergranular corrosion of peened and unpeened Type 304 stainless steel.

dicating that the applied stresses were in fact only partially relieved.

### 5. SENSITIZATION

One pair of U-bend specimens was tested to determine if shot peening will protect sensitized material from stress corrosion cracking. In this test, the U-bend specimens were heated at 1000 °F (538 °C) for 100 h to promote carbide precipitation. After heating, one specimen was peened to an Almen Intensity of 0.008A prior to the SCC test in the MgCl<sub>2</sub>; the other was tested in the as-sensitized condition. The sensitized- and peenedspecimen survived 1004 h in the SCC test, without failing, while the unpeened, as-sensitized, specimen cracked within 2 h.

The stress decay tests discussed in the previous section were tests of sensitized material also since they were heated at 1000 to 1050 °F (538 to 570 °C). However, the specimens in those tests were sensitized after peening, while in this sensitization test, the specimens were heated first and then peened. The results of these tests indicated that shot peening will protect sensitized as well as unsensitized material from stress corrosion cracking.

These scoping studies indicated to Rockwell personnel that severe shot peening could also enhance resistance to intergranular corrosion due to sensitization. In one laboratory test, three coupons were prepared for testing in 10 pct  $HNO_3-3$  pct HF solution; one coupon was peened before subjecting it to sensitizing temperatures [(1 h at 1200 °F (650 °C)]; a second coupon was sensitized first and then peened; the third reference coupon





was sensitized but not peened. Figure 5 shows the results of this test. The peened, then sensitized specimen showed insignificant weight loss after the initial 1/2 h interval (which probably removed surface oxidation on all specimens); in contrast, the weight loss curves of the second and third specimens indicated a continuing corrosion attack. These results indicate that peening prior to exposure to sensitizing temperatures is a deterrent to IGC while peening after sensitizing is ineffective.

#### **B. Weld-Stress Tests**

U-bend specimens are satisfactory for comparing the SCC behavior of materials, but the stress patterns induced by deflection are not typical of those expected in welded structures. To provide more realistic stress patterns and stress levels, a fixture was fabricated in which the specimen would be stressed by solidification of weld metal. The fixture consisted of a rigid U-shaped channel machined from 1 in. stainless steel plate. Test specimens,  $\frac{1}{8}$  by  $\frac{1}{2}$  by 5 in. (0.32 by 1.3 by 12.7 cm), were welded to each side of the "U" and the assembly partially stress relieved at 800 °F (427 °C). The specimens were then stressed by making a full penetration TIG fusion weld pass transversely across the specimen at midlength. Strain gauges attached to one specimen indicated that residual tensile stress levels in order of 24,000 psi (165 Pa) were obtained by this technique. In two MgCl<sub>2</sub>

tests, the stresses were sufficiently high to initiate cracking within 1 h and cause failure within 3 h. In a third test of this design, one of the two weld-stressed specimens was peened on all surfaces to an Almen Intensity of 0.012A using Grade 280 steel shot. The unpeened specimen developed initial SCC cracks in  $\frac{1}{2}$  h with throughcracks in 9 h; the peened specimen was intact after 89 h in boiling MgCl, when the test was terminated.

These tests demonstrated that 1) residual weld stresses can promote SCC and 2) compressive stresses induced by shot peening are sufficient to combat the residual tensile stresses developed by fusion welds and prevent stress corrosion cracking.

#### C. Component Tests

This phase of the study was conducted to establish the practicality of the peening process for sizes and shapes that typify components in a reactor piping system. The scaleup tests were conducted on circumferential butt welds in a  $5^{1}/_{2}$  in. (14 cm) OD, Type 347 stainless steel pipe; weldments of a heavy wall boss to a  $71/_2$  in. (19 cm) OD, Type 321 stainless steel pipe; a  $4^{1}/_{2}$  in. (11.4 cm) Type 316 stainless steel, hexagonal-shaped tube with 20 pct cold work; and a 6 in. (15.2 cm) OD, Type 304 stainless steel pipe, 8 ft (244 cm) in length. These components were not prepared specifically for this study; rather, the sections were selected from available salvage since all were deemed typical of fabricated piping systems and (except for the Type 304 stainless steel pipe section) all contained residual stresses from the fabrication processes. While the fabrication and welding histories are not known, this is not considered to be relevant for the purpose of these tests.

Figure 6 shows the specimens that were evaluated in the SCC tests. Figure 7 depicts the locations of Specimens 1 and 2, sister specimens from the Type 321 stainless steel boss-to-pipe section. Specimen 1 was unpeened, while Specimen 2 was peened on all surfaces at 0.011A Almen Intensity, as determined by the standard Almen test strip. Unpeened Specimen 1 cracked severely in the



Fig. 7-Type 321 stainless steel boss-to-pipe welds.



Fig. 8—Stress corrosion cracks in unpeened Type 321 stainless steel (Weldment Specimen No. 1 after 22 h in  $MgCl_2$  test. No cracks developed in similar peened weldment after 264 h).

MgCl<sub>2</sub> test as shown in Fig. 8. No cracks were detected after the first 4 h; however, after overnight immersion (a total of  $\sim$ 22 h) a multitude of cracks were readily visible adjacent to the weldment in both the heavy-wall boss and the pipe sections. In contrast, the peened specimen was tested for a total of 264 h without visible stress corrosion cracks in the peened surfaces. Two small cracks were detected on the inside diameter surface; however, they initiated on an unpeened edge of the pipe adjacent to the crevice at the boss-pipe interface and are not considered relevant to the test. Another small defect was visible on the peened outside diameter surface of the boss but it had none of the characteristics of a chloride stress corrosion crack and is also believed to be irrelevant to SCC.

Specimens 3, 4 and 5 are circumferential weldments from the  $5^{1}/_{2}$  in. (14 cm) OD, Type 347 stainless steel pipe. Figure 9 shows the cracks that developed adjacent to the weld of the unpeened Specimen 3 during overnight (23 h) immersion in MgCl<sub>2</sub>. Note that the stress pattern indicated by the crack is circumferential and parallel to the weld on one side of the weld head and radially on the other side. In contrast, no crack developed in 120 h of testing on Specimen 4 which was peened on both outside diameter and inside diameter surfaces. Specimen 5 which was  $1/_2$  peened  $-1/_2$  unpeened was also tested for 22 h. No cracks were detectable in the peened half-section after this 22 h exposure; however, cracking was readily visible in the unpeened halfsection. The cracks propagated circumferentially and parallel to the weld until terminating at the interface with the peened surface shown in Fig. 10.

Specimen 6 is a section of a hexagonal, Type 316 stainless steel, pressure tube that had a 15 to 20 pct reduction in wall thickness in its final processing opera-

tion. As for Specimen 5, it was 1/2 peened-1/2 unpeened when subjected to the MgCl<sub>2</sub> SCC test. Cracks were detected in one inside diameter corner of the unpeened section after only 3 h of testing. After testing for 22 h, cracks were present in the flats as well as in the corners of the unpeened section on both inside diameter and outside diameter surfaces. In contrast, no cracks devel-



Fig. 9—Unpeened Type 347 stainless steel pipe weld (Stress corrosion cracks that developed during 23 h in  $MgCl_2$  SCC test. No cracks developed in a similar peened weldment after 120 h).



Fig. 10—Type 347 stainless steel pipe weld showing stress corrosion cracks in unpeened surface after 22 h in  $MgCl_2$  test. (No cracks developed in the peened surface.)



Fig. 11—Type 316 stainless steel hexagonal tube (20 pct C.W.) showing stress corrosion cracks in unpeened surface after 23 h  $MgCl_2$  test. (Cracks were visible in unpeened surface after 3 h in  $MgCl_2$  test. No cracks were detected in the peened surface.)

oped in the peened surfaces during this test. Figure 11 is a photograph of the specimen showing the interface between the peened and unpeened surfaces and the stress corrosion crack present near the corners of the hexagonal tube. The major crack shown in the corner was a through-crack.

# D. Intergranular Corrosion Prevention Tests

For austenitic stainless steels in the solution annealed condition, heating in the temperature range of about 900 to 1500 °F (482 to 816 °C) promotes the preferential precipitation of chromium carbides in grain boundaries and results in the depletion of chromium in the regions adjacent to these grain boundaries. The corrosion resistances of the chromium-depleted grain boundary regions are diminished to the extent that the alloy is susceptible to intergranular corrosion (sensitized). It is theorized that a cold working process, such as shot peening, will break up the surface grains and grain boundaries and provide a multitude of slip planes, twins and dislocations that provide nucleation sites for precipitation of the carbides. Since the carbides precipitate within grains rather than in grain boundaries, the depletion of chromium in continuous grain boundary paths does not occur in the cold worked surface layer and the material is not susceptible to intergranular corrosion. The feasibility of this approach was recognized during the initial scoping studies (Fig. 5). The tests conducted in this phase of the study were intended to verify the earlier results, to further evaluate peened surfaces with respect to those metallurgical factors that contribute to the suppression of intergranular attack, and to define the requirements of an effective cold worked surface.

The efficacy of severe shot peening to suppress IGC is illustrated in Fig. 12. The microstructures shown are typical of peened and unpeened Type 304 stainless steel



Fig. 12—Photomicrographs of peened and unpeened Type 304 stainless steel plate surfaces (sensitized at  $1200^{\circ}F$  (649°C)—1 h and tested for intergranular corrosion in HNO<sub>3</sub>—HF peened with ceramic beads):(a) peened, (b) unpeened.

surfaces after being "sensitized" and tested for intergranular attack in a  $HNO_3$ -HF solution. The cold worked surface of the peened specimen is completely free from attack; in contrast, the unpeened surface is severely and uniformly attacked at the grain boundaries.

Several series of peening tests were conducted in an effort to establish a threshold level of cold work by shot peening. Table IV shows the results of steel shot peening at increasing pressures and times. When peened at 40 psig for 2 min and 70 psig for 2 min (prior to "sensitizing" at 1200 °F (649 °C), there was some intergranular attack in the acid test. When peened at a pressure of 70 psig for times of 4 min or longer, or at 100 psig for 2 min or longer, the cold work induced by the shot peening prevented intergranular attack. These results gave the first indication that a threshold level for peening could be established.

Two similar series of tests were conducted on the same Type 304 stainless steel material, using conventional steel shot for one series and ceramic beads for the

second. These tests were done by MIC at increasing pressures (20 to 80 psig) and increasing coverage. (Note: In commercial peening nomenclature, 100 pct coverage indicates the time required to assure that 100 pct of the surface is bombarded by the stream of shot; 200 pct coverage is about twice and 400 pct about four times that time for 100 pct.) All of the specimens peened with steel shot were found to be susceptible to intergranular attack after sensitizing; however, the degree of attack was not nearly as severe as for the unpeened reference specimen. Metallographic examination revealed that none of the specimens were sufficiently cold worked to prevent grain boundary attack. In contrast, all of the specimens peened with ceramic beads were immune to intergranular attack. Metallographic examination revealed that ceramic bead peening at the lowest intensity (i.e., 20 psig-100 pct) was adequate to severely cold work the surface grains and prevent the surface from being sensitized and susceptible to IGC. The results also revealed that none of the peening parameters (i.e., pres-

	Peening Parameters	Dermitron* Reading	Severn Gauge	Intergranula Corrosion
Steel Shot	Unpeened	4.0	·	Yes
Peened—AI	40 psig- 2 min	3.8	<0.5 pct ferrite	Yes
	70 psig- 2 min	3.5	<0.5	Yes
	100 psig- 2 min	3.1	<0.5	No
	100 psig- 4 min	2.5	1.5-2.0	No
	100 psig- 7 min	0.6	>3.5	No
	100 psig-10 min	0	>3.5	No
Steel Shot	Unpeened	4.0		Yes
Peened-AI	70 psig- 2 min	3.8	0.5-1.0 pct	Yes
	70 psig- 4 min	3.3	0.5-1.0 pct	No
	70 psig- 6 min	2.9	0.5-1.0 pct	No
	70 psig- 8 min	2.0	1.0-1.5 pct	No
	70 psig-10 min	1.0	1.5-2.0 pct	No
Steel Shot	Unpeened	4.0	_	Yes
Peened-MIC	20 psig-100 pct	3.8	<0.5	Yes
	40 psig-100 pct	3.8	<0.5	Yes
	60 psig-100 pct	3.8	<0.5	Yes
	80 psig-100 pct	3.9	<0.5	Yes
	80 psig- 20 pct	3.9	<0.5	Yes
	80 psig-400 pct	3.9	<0.5	Yes
Ceramic Bead	Unpeened	4.0		Yes
Peened—MIC	20 psig-100 pct	3.4	0.5-1.0 pct	No
	40 psig-100 pct	3.0	0.5-1.0 pct	No
	60 psig-100 pct	2.9	0.5-1.0 pct	No
	80 psig-100 pct	2.6	0.5-1.0 pct	No
	80 psig-200 pct	1.4	2.5-3.0 pct	No
	80 psig-400 pct	1.9	2.5-3.0 pct	No

\*Eddy current meter made by Unit Process Assembly, Inc.

#### Table V. Peening Test Summary

Specimen Number	Deaning	Nondestructive Examination		
	Coverage (pct)	(Severn Gauge) (pct ferrite)	Eddy Current	Intergranular Corrosion
Reference	Unpeened	<0.5	40	Severe
1	100 pct	<0.5	40	Moderate
2	200 pct	<0.5	40	Slight
3	300 pct	~0.5	43	None
4	400 pct	0.5-1.0	46	None
5		1.0-1.5	48	None
6		1.5-2.0	51	None
7		2.5-3.5	73	None
8		4.0-5.0	85	None

1. All specimens were peened with 390 grade ceramic beads at 80 psig pressure. The Almen Intensity for all specimens was 0.024A.

2. 100 pct coverage indicates that the time of peening is sufficient to cover the surface; 200 pct coverage doubles the time for 100 pct, etc. Above 400 pct the time of peening was determined by the Severn Gauge.

3. Eddy current meter made by URESCO Company.

sure, time or coverage, Almen Intensity) is sufficient in itself to define a threshold level to prevent IGC.

Table  $\triangledown$  summarizes the results of the final series of IGC peening tests, using ceramic beads. In these tests, the peening was monitored by percent coverage up to 400 pct and by the Severn Gauge, an instrument that is conventionally used to determine the amount of ferrite in austenitic stainless steel weld metal, for longer periods of time. The Severn Gauge indicated <0.5 pct ferrite at 100 pct and 200 pct coverages and about 0.5 pct ferrite at 300 pct coverage. Note that IGC was prevented at 300 pct coverage, thus the indicated threshold level for 390 grade ceramic beads at 80 psig is 300 pct coverage or 0.5 pct ferrite. The use of peening control techniques, including the Severn Gauge, will be discussed in the next section.

As previously stated, an 8 ft (244 cm) length of 6 in. (15.2 cm) OD, 0.250 in. (0.64 cm) wall, Type 304 stainless steel pipe was peened on outside diameter and inside diameter surfaces using commercial automated equipment. The measured Almen Intensity for the peening operation was 0.012. It was intended that the peening process would be controlled with the Severn Gauge but this proved to be unsuccessful. At 100 pct coverage (2 passes through the peening machine), the amount of ferrite formed was <0.5 pct, the minimum readout on the Severn Gauge. Peening was continued to coverages of 200 pct (4 passes), 300 pct, 400 pct, 800 pct, and ultimately to 1400 pct, without detectable levels of ferrite being formed. Subsequent metallographic examinations and corrosion tests confirmed that the cold working induced by the peening was insufficient to produce 0.5 pct ferrite or to prevent intergranular attack regardless of peening time (coverage). This deficiency of cold work is attributed in part to the use of a smaller size (170 grade) shot instead of the larger sizes used in previous tests. It is believed that acceptable cold worked surfaces can be attained using larger shot and increased intensity. This is substantiated by the fact that the inside diameter surfaces, which were peened to 400 pct coverage (via a wand that was passed through the pipe), were more highly cold worked than the outside diameter surfaces. The amount of cold work on the inside diameter surface was not sufficient to form 0.5 pct ferrite, but it was enough to prevent intergranular attack in the corrosion tests.

#### **IV. DISCUSSION**

This investigation demonstrated both the feasibility and the practicality of preventing stress corrosion cracking and intergranular corrosion in Type 304 stainless steel and probably in any similar austenitic stainless steel. It was found that resistance to SCC is achieved over a wide range of shot peening conditions, shot sizes and materials; is effective on sensitized as well as unsensitized material; and is not negated by prolonged heating at stress relieving temperatures to 1050 °F (566 °C). The effectiveness of shot peening for preventing this type of corrosion is probably dependent, in the main, on achieving complete surface coverage rather than on any particular processing parameter. This is illustrated by the fact that there were no failures due to stress corrosion cracking in any peened surface, regardless of the

peening condition. Of the 46 U-bend specimens tested, a few did in fact crack; however, the cracks initiated in unpeened surfaces, as in those specimens with intentional peening coverage defects or in unpeened locally stressed areas of the specimen legs that were remote from and unrelated to the applied stresses in the U-bend area. The latter premature failures were subsequently eliminated by peening all surfaces of the specimen. As previously mentioned, unrelated cracks developed in the peened surfaces of one welded component (Specimen 2, Fig. 6). One was probably due to SCC; however, it developed in an unpeened, crevice area at a weld joint; the second is not attributed to SCC since it was a single, uniformly arced crack that was not at all characteristic of the classical branching pattern of chloride stress corrosion. In spite of these few observations, the test demonstrated that peening, even if not an infallible preventive, is a strong deterrent to SCC. This is illustrated by the significantly longer times for cracks to develop and by the fact that the peened surface served as a crack arrester for those cracks that developed in adjacent unpeened areas.

With respect to preventing intergranular corrosion, it is necessary to control the peening process by means other than the conventional peening intensity and coverage. The surface grains must be cold worked sufficiently to prevent the precipitation of carbides along continuous paths if subsequently exposed to sensitizing temperatures. The capability of the shot peening process to attain this degree of cold work is illustrated in Figs. 13 and 14. Figure 13 shows an electron micrograph of an unpeened, Type 304 stainless steel surface after sensitizing. Note the massive carbide particles that precipitated along the grain boundary; since the alloy adjacent to each particle is depleted in chromium (due to the forma-



Fig. 13—Electron micrograph of unpeened Type 304 stainless steel, sensitized for 24 h at 1250 °F (676 °C). (Carbide particles have been precipitated along grain boundary.)

J. MATERIALS FOR ENERGY SYSTEMS



Fig. 14—Electron micrograph of shot-peened surface of Type 304 stainless steel plate, after sensitizing 24 h at 1250 °F (676 °C). (Carbides are precipitated randomly throughout matrix without preference for grain boundaries.)

tion of the chromium carbide), the net result is a continuous path of a noncorrosion resistant material that is susceptible to intergranular attack. In contrast, Fig. 14 shows an electron micrograph of a peened surface exposed to the same sensitizing treatment. Here the carbides have precipitated as small, randomly dispersed particles throughout the grain without preference for grain boundaries. In this condition, there are no continuous paths of chromium-depleted alloy and intergranular attack cannot take place.

The feasibility of both magnetic and eddy current techniques for controlling the amount of cold work on a peened work piece has been demonstrated (Tables IV and V); however, further work is required to define their full capabilities and limitations. To date, the application of the magnetic technique, using a Severn Gauge, shows promise. The Severn Gauge uses a series of magnets that are calibrated in "pct ferrite," and is conventionally used to determine the amount of ferrite in austenitic stainless steel weld metal. This principle can be applied to monitor peening since ferrite may be formed by a martensitic reaction during the cold working of the surface grains. Using the Severn Gauge, it was shown that peening developed up to 7.5 pct ferrite in Type 304 stainless steel. Development tests also demonstrated that the presence of 0.5 pct ferrite in a peened surface is indicative of sufficient cold work to prevent intergranular attack in Type 304 stainless steel. This approach has its limitations in that 1) it will also respond to the ferrite in any peened welds, and 2) it cannot be applied to alloys other than the 18-8 types of stainless steels that are subject to the martensitic reaction.

The eddy current technique can be adapted to monitor shot peening since it responds to changes in electrical properties. Using an unpeened standard of the same



Fig. 15-Results of eddy current (Dermitron) inspection of various shot-peened surfaces.

material and thickness, an eddy current instrument will indicate changes in the electrical properties induced by the cold worked surface grains. An example of one such correlation is shown in Fig. 15. Using a commercial eddy current instrument designed to measure the thickness of coatings on metals, a general correlation between the eddy current response with the time of peening was developed for 1/4 in. (0.64 cm)-thick plate material in the HRAP (hot rolled, annealed, pickled) condition. For this technique, a new calibration curve has to be developed for each material and condition. Tests to date have indicated that the eddy current is very sensitive to local variations in the peened surface and as such, considerable judgement must be exercised in interpretation of results.

#### **V. CONCLUSIONS**

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Shot peening can prevent stress corrosion cracking by imposing compressive stresses on the surface of the work piece. In general, adequate compressive stress levels were attained by 200 pct surface coverage, *i.e.*, twice the time required to peen 100 pct of the surface area.

Controlled shot peening can prevent intergranular corrosion due to sensitization. Peening must be sufficient to severely cold work the surface grains.

The feasibility of shot peening to prevent corrosion of austenitic stainless steels has been established. The practicality of shot-peening processes, equipment and control techniques, needs to be demonstrated for industrial applications.

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