3ND-P870-2481

UNCLASSIFIED

TECHNICAL REPORT

U. S. NAVAL APPLIED SCIENCE LABORATORY

NAVAL BASE

BROOKLYN 1, NEW YORK

EFFECT OF PICKLING ON NOTCH-TOUGHNESS AND SURFACE PITTING OF HY-80/100 TYPE STEEL PLATE

LAB.PROJECT 9300-1, PROGRESS REPORT 2
SR 007-01-01

13 Jan 1965

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SUMMARY

The objective of this investigation was to develop a uniform and improved pickling procedure for the descaling of HY-80/100 type steel plate. The principal governing criteria were (a) maximum depth of pitting and (b) effect on notch-toughness as evaluated by the Navy tear test.

Results indicate the following:

a. HY-80/100 steel plate may be pickled in the standard solution for time intervals up to two hours without adverse effects on the material provided that there is at least a 48 hour interval before fabrication.

b. Windrowing or deep furrowing of plate surfaces observed after removal of the steel from the acid bath is associated with the prior mill processing and not the chemical action of the pickling solution.

The following modifications in the pickling procedure of reference (c) are recommended for HY-80/100 type steel:

a. Pickling times up to two hours should be permitted provided that there is at least a 48 hour interval before fabrication (24 hours currently specified).

b. For intervals of less than 48 hours, rinse time after pickling should be increased to 30 minutes or the plate should be heated at 200°F for at least four hours.

Consideration should be given to modification of the inhibitor specification (reference (n)) to screen out those inhibitors which might have embrittling effects.

TABLE OF CONTENTS

FIGURES (Cont'd)

TABLES

ADMINISTRATIVE INFORMATION

Ref: (a) NAVAPLSCIENLAB Program Summary dtd 1 Nov 1963 for SR 007-01-01, Fabrication of High Strength Structural Steel Alloys
(b) BUSHIPS ltr R007-01-01, Ser 634B-586 of 10 Jul 1963
(c) BUSHIPS Technical Manual 250-000-19 of 15 Jul 1962
(d) MIL-S-16216G (SHIPS)
(e) BUSHIPS ltr L8/MIL-S-16216(343) Ser 343-121 of 30 Apr 1958
(f) NAVSHIPYDNYK MATLAB Project 5498, Progress Report 2, "Pickling of Medium, High Tensile and Special-Treatment Steel Plate" by E.A. Imbembo and F.G. Ginsberg, dtd 10 Feb 1956
(g) NAVSHIPYDNYK MATLAB Project 5498, Final Report, "Pickling of Special-Treatment Steel Plate" by F. Ginsberg and I. Geld, 1 Jul 1959
(h) NAVSHIPYDNYK MATLAB Project 5152 and 5152-1, Final Report, "Effects of Variations in the Geometry of the Tear Test Specimen as Applied to the Evaluation of Notch-Toughness of Ship Plate Steels" by E.A. Imbembo and F. Ginsberg, 21 May 1956
(i) "A Method of Evaluating Transition from Shear to Cleavage Failure in Ship Plate and Its Correlation with Large-Scale Plate Tests", N.A. Kahn and E.A. Imbembo, The Welding Journal, 27 (4), pp 169S-186S (Apr 1948)
(j) "Notch-Sensitivity of Ship Plate - Correlation of Laboratory-Scale Tests With Large-Scale Plate Tests", N.A. Kahn and E.A. Imbembo, ASTM Special Technical Publication No. 87, pp. 15-52 (1949)
(k) "Further Study of Navy Tear Test", N.A. Kahn and E.A. Imbembo, The Welding Journal, 29 (2), pp 84S-96S (Feb 1950)
(l) NAVSHIPYDNYK ltr 981:FG:sk, L18/metallurgy, Lab. Project 5918-1, Progress Report 2 of 21 Aug 1959
(m) NAVSHIPYDNYK ltr 981:FG;mm, Lab. Project 5918-1, Progress Report 3 of 9 May 1960
(n) Federal Specification O-I-501b dtd 2 Jan 1964, Inhibitors, Pickling (for Use with Sulphuric Acid)

1. Authorization to conduct this investigation which is a part of the continuing program on fabrication of high strength steel alloys, is contained in reference (a). The work was conducted along the lines indicated in reference (b).

ACKNOWLEDGMENT

2. This project was carried out by the Metallurgy Branch, with the joint effort of the Inorganic Chemistry Branch of the Physical Sciences Division, under the respective supervision of Messrs E.A. Imbembo and W.L. Miller. This work is a facet of the Laboratory's High Strength Steel Program which is being conducted under the overall direction of Mr. I.L. Stern. Mr. J.J. Gabriel made valuable contributions in surface pit depth measurements. The mechanical tests were performed by the Mechanics Branch under the supervision of Mr. H.V. Cordiano.

The interest and support of personnel of the Bureau of Ships, Mr. T. Dawson, cognizant engineer, Code 634B and Mr. G. Sorkin, Program Manager, Code 341A are also appreciated.

3. Identification of the mills, including heat numbers of the plate samples investigated, is given in Table 9. This table may be deleted at the discretion of the Bureau. Unless otherwise directed within 30 days, the Laboratory will scrap the remaining sample material.

BACKGROUND

4. The Bureau became concerned with the problem associated with HY-80/100 plate pickled in accordance with the requirements of reference (c) because of the following considerations:

a. Deep surface irregularities have been observed on HY-80 plate after descaling with the reference (c) solution. An example illustrating this condition is shown in Figure 1. Severe windrowing which is essentially a series of deep, wide pits contiguous to each other may be observed mainly in the left hand portion of the lower photograph. In contrast, the same areas in the upper picture before pickling show little evidence of this condition. Two possible sources may be responsible for the pitting illustrated in Figure 1, as follows:

(1) Mill processing which may introduce pitting such as windrowing or individual, small diameter but relatively deep pits, either isolated or in clusters, by the mechanism of rolling-in mill scale.

(2) Chemical action associated with galvanic or electrolytic cell formation during the initial stages of acid descaling. Possible sources of these cells are penetration of the mill scale through surface cracks wherein the base metal acts as an anode and the scale a cathode, or areas in which lightly and heavily scaled portions are adjacent to each other. In the latter case, the thinner scale may be chemically removed first, the exposed metal surface of which may then act anodic to the adjacent, more heavily scaled area. Such a situation has been thought to cause excessive pitting and reduction in plate thickness in the thinly scaled areas, particularly if immersion time in the bath is excessively long.

b. In pickling HY-80 plates, immersion times up to six hours have been reported. It is possible that pickling smut which develops on HY-80 plate surfaces can be mistaken for scale and thus motivate extended immersion periods. Such long pickling times increase the probability of hydrogen embrittlement. Associated matters of concern are the alleged possibility of causing the poor surface appearance described above and excessive thickness loss.

5. Excessive surface pitting and windrowing are contrary to the requirements of references (d) and (e). They specify that the depth of rolled-in scale and pits or windrowed condition shall not exceed 0.015" maximum; isolated, individual pits not over 0.030" deep are permitted provided they do not reduce the thickness of the plate below the specified minimum.

6. Information was therefore needed to determine whether the specified pickling solution and procedures are responsible for the conditions noted and, in addition, whether any deleterious effects (hydrogen embrittlement) are found in the notch-toughness of HY-80/100 type steel as a result of pickling.

OBJECT

7. The objective of this investigation was to develop a uniform and improved pickling procedure for the descaling of HY-80/100 type steel plate. The governing criteria were as follows:

a. The maximum depth of the surface pitting.

b. The notch-toughness properties as evaluated by the Navy tear test.

c. Time for complete removal of scale as defined by ohmmeter readings.

d. Loss in plate thickness.

DESCRIPTION

8. Tests and experiments covered in the current investigation were performed on material taken from the following samples of plate:

a. Plate Code S: One (1) HY 80 plate, 96"x240"x1" thick

b. Plate Code K: One (1) HY-80 plate, 96"x120"x1" thick

c. Plate Code 93G: One (1) HY-80 plate, 14"x99"x1" thick. This plate remained from a previous investigation, Lab.Project 5918.

9. The mill compositions and static tensile properties of the sample plates are shown in Table 1, along with the requirements of the current specification, MIL-S-16216G (SHIPS).

EXPERIMENTAL PROCEDURE

10. The composition of the pickling solution employed conformed to the requirements of reference (c). This bath which shall be henceforth called the standard solution consisted of the following:

Sulphuric acid (66°Be, A.C.S. grade)-5% by volume
Code X inhibitor -0.21% by volume of concentrated sulphuric acid *
Sodium chloride - USP, 1.5% wt/volume

* Code X is identified in Table 9

To this solution, 2.5% wt/volume of iron was added by dissolving HY-80 steel, simulating a "naturally aged" bath which also contained solute alloying elements and tramp impurities. A new solution of this composition was utilized for each pickling operation. The standard procedure specifies a maximum immersion time of 1 1/4 hours at 175°F ± 5°F followed by a two minute rinse in water at 175°F.

11. Prior to pickling, the test specimens were degreased with toluol and all machined areas were masked with acid resistant lacquer, exposing only the mill scale.

Pit Depth Determination and Surface Appearance

12. Test panels, 6"x10"x1" thick, were immersed in the standard solution at a temperature of 175°F. For plates S and K, samples were immersed for 1 1/4, 2 and 4 hours. For 93G, two test specimens were pickled; one for 1 1/4 hours and the second for 4 hours. Using a calibrated microscope, pit depths were measured first by focusing on the bottom of the pit and then on a strip of adhesive paper placed adjacent to the pit under consideration at its upper irregularly shaped edges. By this means, a plane of reference was established. The difference between the two readings minus the previously determined thickness of the paper constituted the pit depth. Areas for measurement of relatively deep, intermediate and shallow pits were selected at random with the aid of a 3X magnifying glass.

13. Plate S had been manufactured at the mill under special conditions in an attempt to develop a thick mill scale for investigational purposes. The procedures employed by the mill were as follows:

a. Slab was not covered with burlap prior to going through the scale breaker.

b. No water spray was applied to slab surfaces while going through the scale breaker.

c. Final plate was maintained at the austenitizing temperature about 50% longer than usual.

After the scale was removed by pickling at the Laboratory, the plate surfaces showed considerable windrowing with deep pits (see Figure 1). However, some of the irregular surface appearance was also visible in the unpickled state where the scale has spalled off. Thus, it became necessary to ascertain whether pickling had caused the wide deep pits in such profuse amounts or whether the defects were there initially, before pickling, but concealed by mill scale. To resolve the question, one of the 6"x10"x1" thick panels from plate S was descaled fully by sandblasting for 1 1/2 minutes on each surface so that such a determination could then be made. It should be noted that plate S was not rolled under normal mill conditions and the resulting plate surfaces should not be considered as typical of the manufacturer's product.

Notch-Toughness Properties (Tear Tests)

14. The effects of pickling on notch-toughness properties (hydrogen embrittlement) were evaluated by means of the Navy tear test. This method was employed since previous work reported in references (f) and (g) demonstrated that this test method was an effective indicator of hydrogen embrittlement of special-treatment steel (similar in composition and strength to HY-80/100) when subjected to acid pickling. In these studies, adverse effects on notch-toughness were noted principally by a change in fracture appearance and a decrease in the energy value to propagate fracture. In the current investigation, the total energy (initiation plus propagation) was also considered.

15. The test procedure used by the Laboratory in processing the various tear specimens during pickling is summarized in Table 2. The tear specimens were taken in the longitudinal rolling direction and tested in full plate thickness. The conventional tear specimen employs a nominal 2" fracture length. Due to limitations in load testing capacity, this was reduced to 1 1/2" for specimens taken from plates K and 93G and 1" for the Code S samples. The work which validated the 1 1/2 inch or 1 inch fracture length tear specimen is described in reference (h). The experimental procedure and method of evaluating results of tear tests have been fully described in references (i), (j) and (k).

16. For a particular sample plate, the tear specimens were tested inthe prime and pickled conditions at a selected temperature. This was taken as the lowest temperature which would consistently result in ductile behavior in the prime condition.

Scale Removal

17. The pickled test panels were checked for remaining mill scale on both surfaces after specific time intervals in the pickling solution by means of an ohmmeter with light to moderate pressure on the test prods. Metal surfaces with a resistance of less than 1/2 ohm are indicative of complete scale removal.

Scale Thickness Determination

18. Thickness of mill scale for each surface of the sample plates was determined by standard metallographic techniques at 250X.

Thickness Loss Due to Pickling

19. Loss in plate thickness was determined by means of the following formula after approximate micrometer measurements on the respective panels in the unpickled and pickled condition:

$$L = P - (2S+A)$$

L = average thickness loss
P = average thickness of prime plate (with scale)
A = average thickness of pickled panel
S = average scale thickness, each face

RESULTS AND ANALYSIS

Pit Depths and Surface Appearance

20. Figures 2 and 3, representing plates K and 93G, show satisfactory surfaces after pickling for 4 hours. Except for some isolated pits, which are not excessively deep, these surfaces are relatively smooth. On the other hand, the pickled surface of Plate S, shown in Figure 1 (also pickled for 4 hours), illustrates a non-acceptable windrowed condition, consisting of wide deep pits. As previously indicated, it should be noted that the surface condition of this plate is not a normal production product. The surface was obtained in an effort by the mill to build up the scale thickness of the material (see paragraph 13) in compliance with a request by the Laboratory.

21. Referring to Figure 4, it may be observed that the sandblasted panel contains a number of parallel rows of "ridge and valley" effects. Part of this condition may be observed on the "as received" surface of the material (prime plate, non-sandblasted), particularly in areas where the mill scale had spalled off. After complete removal of the mill scale by sandblasting, the full extent of windrowing was revealed.

22. A tabulation illustrating the distribution and range of pit depths for each of the sample plates after pickling or sandblasting is given in Table 3. Based upon these data, the following observations are made:

a. Plates K and 93G gave pit depth values which fall within the requirements of references (d) and (e). Microscopic measurements of these samples indicated that the higher values and particularly those over .015" were rare in number and well isolated. Extending the immersion time to 4 hours did not result in pits substantially deeper than those obtained after 1 1/4 hours.

b. Plate S showed an unsatisfactory surface condition since it has large deep pits contiguous to each other so as to form a severely windrowed condition. This plate was unacceptable with respect to the surface quality requirements of references (d) and (e). The maximum pit depths were excessive and occurred in large clusters. The 2 hour pickled panel showed the greatest pit depth, namely 0.074". This suggests that the 0.074" value was derived from pits which were initially deeper in the prime plate than those in the 1 1/4 and 4 hour samples.

c. The pit values of the sandblasted sample from plate S were on the same order of magnitude as those in the pickled panel taken from plate S. This observation coupled with the discussion of Figure 4 in paragraph 21 indicate that the pitting described in paragraph 4a did not result from the action of the pickling bath but was formed during mill rolling.

Notch-Toughness Properties (Tear Tests)

23. Results of tear tests are given in Tables 4, 5 and 6. In assessing these data, variations in average energy values up to approximately 15% between pickled and prime plate conditions were not considered significant since such differences may be encountered in tear test results.

24. A discussion of the data follows:

a. Table 4 - There were no significant differences in the tear test properties of plate K between the prime and pickled conditions (1 1/4 or 4 hours pickling) followed by a 24 hour age at 15°F; the 24 hour layover is required by reference (c), without specific reference to temperature.

b. Table 5 (1 1/4 hour pickle) - In the case of plate 93G, the results after pickling for 1 1/4 hours and a 2 minute rinse (batch 1) showed that the notch-toughness properties were not adversely affected.

c. Table 5 (4 hour pickle) - The 4 hour pickling period for plate 93G with a nominal 2 minute rinse, without and with a 24 hour aging period prior to testing produced marked embrittlement with respect to fracture appearance and energy-to-propagate value (batch 2 and 3). This is also reflected in the total energy. The data indicated a noticeable improvement due to the 24 hour aging. Extension of the time of immersion in the rinse water at 175°F from 2 minutes to 30 minutes after pickling (batch 4) resulted in sufficient recovery of the properties to be considered satisfactory. In order to determine whether the 24 hour aging period after pickling could be eliminated with the 30 minute rinse, an additional batch of specimens was tested (batch 5) without aging after pickling. Results indicated that with the 30 minute rinse there were no adverse effects on notch-toughness with elimination of aging.

d. Table 6 (1 1/4 hour pickle) - For plate S, the 1 1/4 hour pickling period with either a 2 minute (conventional) or 30 minute immersion in the rinse water at 175°F prior to aging (batches 1 and 2, respectively) indicated a loss of 12.5 per cent energy-to-propagate value when compared to prime plate results, which is considered satisfactory.

e. Table 6 (4 hour pickle) - Each of the results for the 4 hour pickling period, batches 3 and 4, indicated an average loss in energy-to-propagate value of 16 per cent for the pickled plate, which is not considered satisfactory. Neither aging for 24 hours with the conventional 2 minute rinse or immersion of the pickled material in a rinse bath at 200-205°F for 30 minutes without aging was sufficient to effect a fully satisfactory recovery of properties. The above enery-to-propagate value for the pickled plate may be considered of borderline acceptability since it slightly exceeds the permissible loss of 15 per cent for this property established for the test procedure. Attention is invited to the fact that greater numbers of specimens were run in some of the tests due to the badly windrowed condition of the surfaces and the Laboratory considered that, in these cases, the conventional number of specimens might not be indicative of the true behavior of the plate.

25. Discounting the marginal behavior of plate S which had an unusual and unacceptable surface condition, the above findings indicate that for pickling times appreciably in excess of 1 1/4 hours, there is some danger of inducing embrittlement effects which can be obviated by extending the time before fabrication to 48 hours; if the 48 hour aging is not feasible, the rinse time after pickling should be increased from 2 minutes to 30 minutes to produce the same effects. In this connection, it is considered that the practice of preheating for some fabrication operations at about 200°F for periods of 4 hours or more would result in substantially the same effects as either of the two treatments noted above.

26. The work reported to this point was concerned with 1" thick HY-80 plate. Previous pickling studies performed by the Laboratory on 2" thick HY-100 plate are summarized in Table 7. This material represented experimental production heats, a description of which is given in references (l) and (m). At the time, the question of surface pitting effects was not a matter under consideration. The data which are based on a 1 hour pickling time indicate no adverse effects in plate A with a 2 minute rinse and 24 hour aging. In the case of plate AA, deleterious effects by pickling were indicated by the energy values obtained with a 2 minute rinse and 24 hour aging; the properties, however, were satisfactorily restored by a 48 hour aging treatment following the 2 minute rinse. While these data are based on a 1 hour pickle, the substantial improvement due to the 48 hour aging, suggests that the extension of the aging time from 24 to 48 hours might serve as an alternative to a 30 minute rinse, particularly if the

maximum time of pickling is limited to a practical value of 2 hours. Pickling times in excess of 2 hours are not considered necessary to remove normal scale (as will be noted below), are not economical, and could decrease the margin of safety with respect to hydrogen embrittlement. In addition, it should be noted that the aging was conducted at 15°F (for reason indicated in Table 2) but in actual practice the ambient temperature would most probably be well above 15°F, thus favoring hydrogen evolution.

27. With respect to the notch-toughness behavior of plate S, the following comments are considered pertinent: This material had a very unsatisfactory surface condition - windrowing (deep wide pits) developed at the mill during manufacture (see Figure 1) which resulted in "peaks and valleys" configurations. This plate gave the greatest variation in pickling time to remove scale over the 6"x10" test panel surface with the smallest interval occuring at the depressions; approximately 30 minutes was required to descale the "valleys" as compared to 80 minutes for the "peaks". These time intervals were determined by ohmmeter measurements. Since descaled metal was exposed in the "valleys" prior to the "peaks", galvanic or electrolytic cells were set up initially with the descaled "valleys" as anodes and the scaled "peaks" as cathodes. This in turn increased hydrogen generation which provided a greater potential for embrittlement. Following complete descaling after 80 minutes, the generation of hydrogen continued as a result of acid attack on the bare metal but at a slower rate. In connection with plate S, the increase in actual surface area due to windrowing enhances the possibility of greater hydrogen absorption compared to K and 93G. This effect is amplified when the immersion time in the pickling bath is extended to 4 hours where hydrogen absorption becomes critical.

28. Test results reported herein are based on the use of Code X inhibitor, which conforms to the requirements of reference (n). This specification provides for the evaluation of inhibitors principally by rate of scale removal and hydrogen evolution. No provision is made for determining hydrogen absorption into the steel and embrittlement caused thereby. It is known that different commercial inhibitors vary considerably with respect to the amount of hydrogen absorbed by the steel during pickling. It is conceivable that other inhibitors which meet the requirements of reference (n) may however cause excessive hydrogen embrittlement. The conclusions and recommendations made herein therefore refer only to pickling processes with code X inhibitors.

Scale Removal

29. For the samples investigated, complete descaling was accomplished in the following order: plate K, 18 minutes; plate 93G, 44 minutes; and plate S, 80 minutes. Completness of descaling was determined by resistance measurements. This wide variation in pickling time may be considerably influenced by the surface condition of the plate. Relative evaluations of surface condition may be

made from comparative studies of Figures 1, 2 and 3. Plates K and 93G are considered satisfactory with respect to surface while S is not. The order of merit correlates with the reported times for complete descaling. Considering the "galvanic cell" theory discussed in paragraph 27, it is quite possible that some of the hydrogen generated by the electrolytic cells could partially blanket the metal surfaces from the action of the acid bath and thereby increase the pickling time. The amount of this increase would depend upon the extent of windrowing. The smoothest plate, K, would provide fewer galvanic cells and generate less hydrogen. In addition, the mill scale on plate K is considerably thinner.

Mill Scale Thickness

30. The approximate maximum and minimum thickness of mill scale for the 1" thick sample plates are shown in Figure 5. Plates S and 93G were produced by one manufacturer while plate K represents a second mill. Based on the average of a considerable number of scale thickness measurements, it was established that all plates tested had a thicker scale on one surface than on the other. However, the surface having the thicker scale in plate K did not approach the corresponding thicker surfaces of plates S and 93G. Approximate total thickness values for mill scales are presented in Table 8. Figure 6 illustrates the surface scale appearance and scale thickness difference between typically spalled and unspalled adjacent areas of HY-80 material. Plate S was used as the example.

Thickness Loss Due to Pickling

31. Thickness losses, as a result of pickling for various immersion times, were determined for the 1" thick plate samples. A tabulation of results is given in Table 8. On the basis of these data, the following comments are made:

a. The loss in plate thickness after pickling was about the same for 1 1/4 hours as it was after 4 hours for plates S and K. Plate 93G showed a slightly higher loss after 4 hours of pickling as compared to 1 1/4 hours.

b. Taking into account the total mill scale removed from each of the samples in pickling, the results indicate that the amount of clean metal dissolved in the acid solution, as measured by thickness loss, was very small or in the case of plate K negligible. The higher metal loss of plate S in the 1 1/4 hour pickle, as compared to plates K and 93G, is attributed to its surface condition which contributed to the formation of electrolytic cells, as described previously.

c. In the case of plate S in which the apparent metal loss after 4 hours of pickling is slightly less than that for the 1 1/4 hour period, the difference is probably due to the irregular surfaces which made precise measurements difficult.

CONCLUSIONS

32. On the basis of the data presented herein for HY-80/100 steel plate, the following conclusions are made:

a. Plate may be pickled in the standard solution for time intervals up to two hours without adverse effects on the material provided there is at least a 48 hour interval before fabrication.

b. Although no pit depth or thickness loss measurements were made on the HY-100 plates, it is considered that no significant differences from HY-80 would occur with respect to these parameters.

c. Where windrowing or deep surface pits are noted upon removal from the pickling bath, the unsatisfactory surface is not to be construed as caused by attack of the pickling solution. It is an indication of a pre-existing condition which had not been observed because of masking mill scale.

d. Pickling time in the pickling solution may be increased from 1-1/4 (now specified) to 2 hours. Pickling times in excess of 2 hours are not considered necessary to remove normal scale, are not economical, and could decrease the margin of safety with respect to hydrogen embrittlement. In abnormal plates, scale remaining after a 2 hour pickle, should be removed by mechanical means.

e. Significant embrittlement was observed for 4 hour pickling times. This embrittlement was significantly reduced wth a 30 minute rinse.

f. The data do not provide information on the hydrogen inhibiting characteristics of approved inhibitors other than Code X; other inhibitors conforming to reference (n) should be examined to assure that embrittlement effects are no greater than those shown by Code X. Evaluation procedures such as the Navy tear test may be used for such a purpose.

RECOMMENDATIONS

33. It is recommended that the pickling procedure of reference (c) for HY-80/100 type steel be modified to permit pickling times up to 2 hours provided that there is at least a 48 hour interval before fabrication (24 hours currently specified). While no data are available as to effects of pickling on plates over 2" in thickness, this recommendation is considered applicable since it is assumed that the heavier plates will be heated during fabrication to temperatures in excess of 250°F for 4 hours or more.

34. In the unusual case where fabrication is required before 48 hours, then either precaution (1) or (2) listed below should be used:

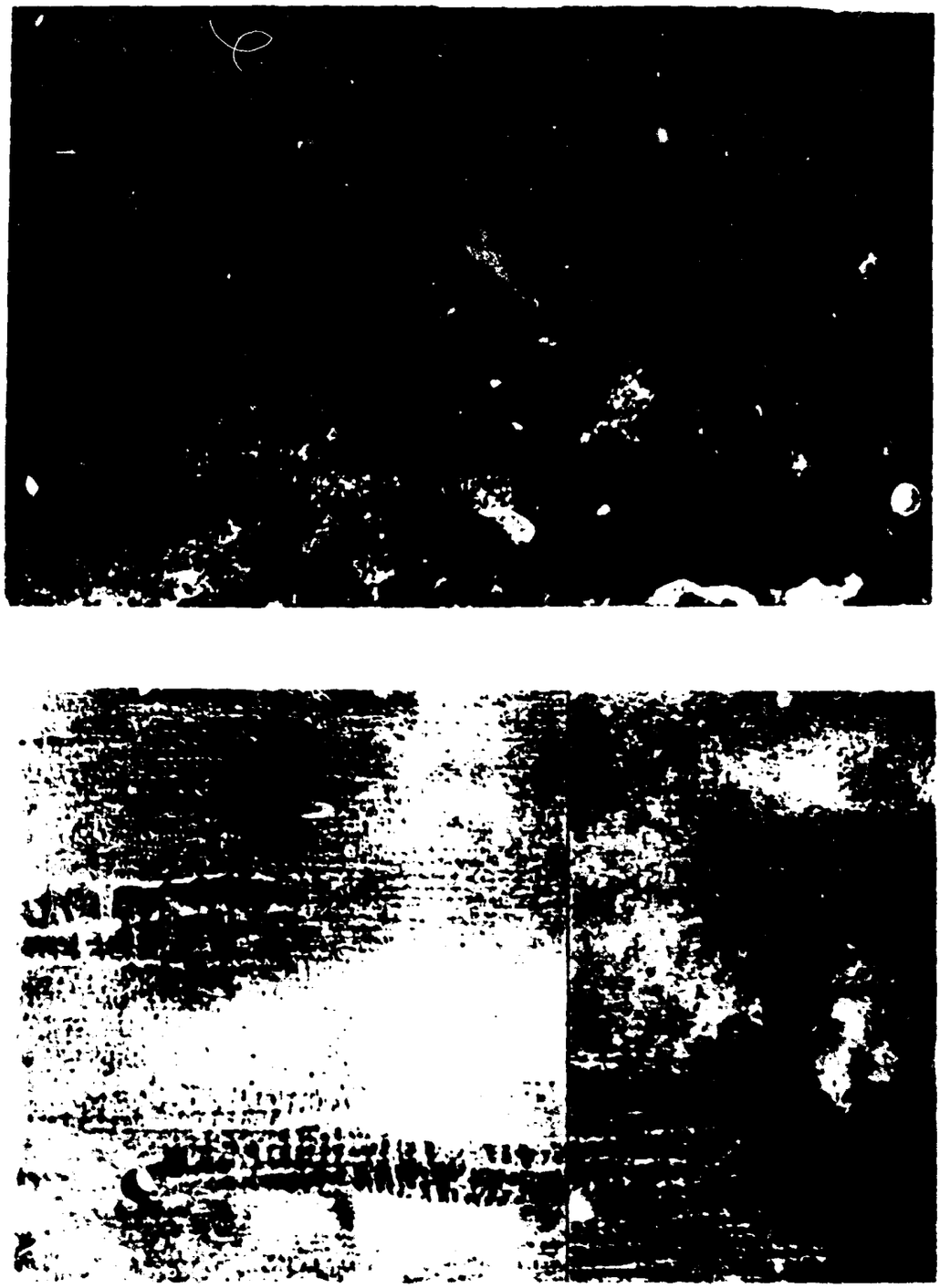
(1) Preheating at temperatures of 200°F or higher for a minimum of 4 hours.

(2) Time in rinse bath should be increased from the currently specified 2 minute period to 30 minutes.

35. Consideration should be given to modification of inhibitor specification to eliminate inhibitors which might have adverse effects on mechanical properties.

FUTURE WORK

36. This report concludes planned work on the pickling of HY-80/100 type steel. Work planned for Fiscal Year 1965 is being directed at studies on pickling of HY-150 steel with a view of determining whether the modified pickling procedures can be used without adverse effects on material.



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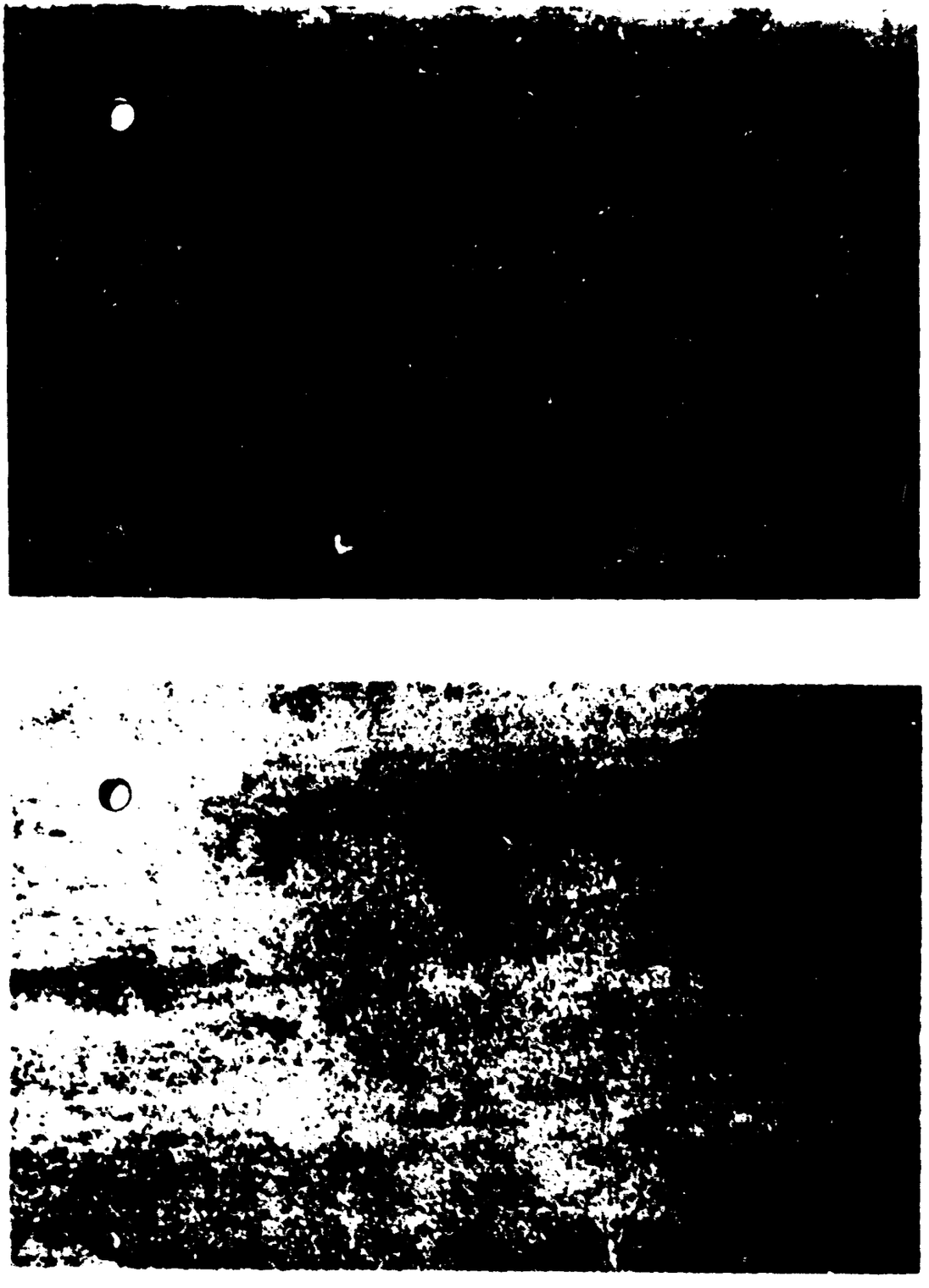
Lab.Project 9300-1
Progress Report 2
Photo No. L-19780-1

Figure 1 - Photomacrographs of a 6"x10"x1" Thick Section of HY-80 Plate Showing Unpickled (Upper) and Pickled (Lower) Plate Surface
Plate S - Pickling Time: 4 Hours
Approx. 3/4X



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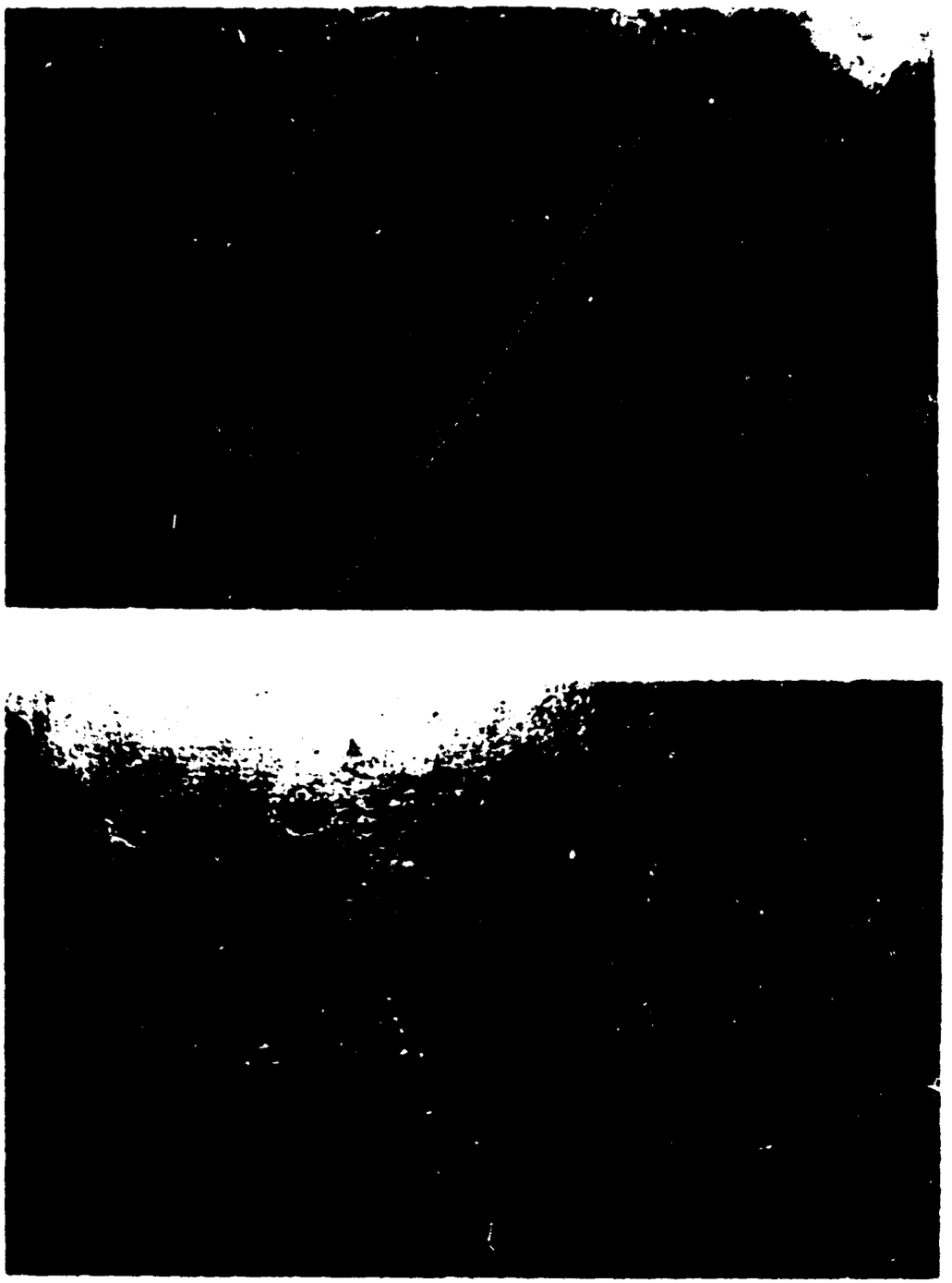
Lab. Project 9300-1
Progress Report 2
Photo No. L-19780-2

Figure 2 - Photomacrographs of a 6"x10"x1" Thick Section of HY-80 Plate, Showing Unpickled (Upper) and Pickled (Lower) Plate Surface
Plate K - Pickling Time: 4 Hours
Approx. 3/4X



U.S. Naval Applied Science Laboratory

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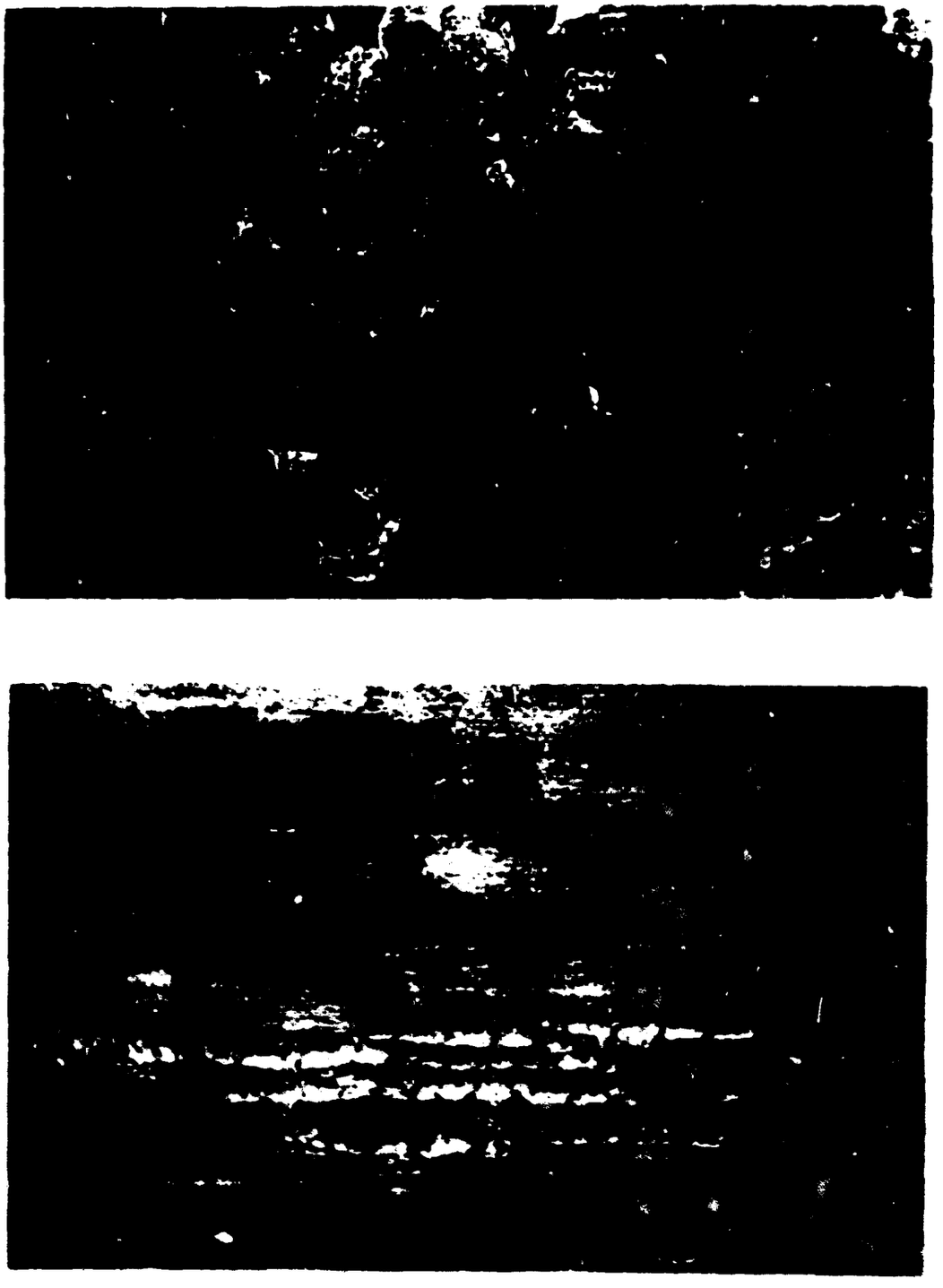
Progress Report 3
Photo No. [illegible]

Figure 3 - Photomacrographs of a 6"x10"x1" Thick Section of HY-80 Plate, Showing Unpickled (Upper) and Pickled (Lower) Plate Surface
Plate 93G - Pickling Time: 4 Hours
Approx. 3/4X



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Lab. Project 9300-1
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Photo No. L-19780-4

Figure 4 - Photomacrographs of a 6"x10"x1" Thick Section of HY-80 Plate, Showing Unpickled (Upper) and Sandblasted (Lower) Plate Surface
Plate 5 - Approx. 3/4X

Plate S - .0055 in.

Plate S - .0015 in.

Plate K - .003 in.

Plate [illegible] - .0018 in.

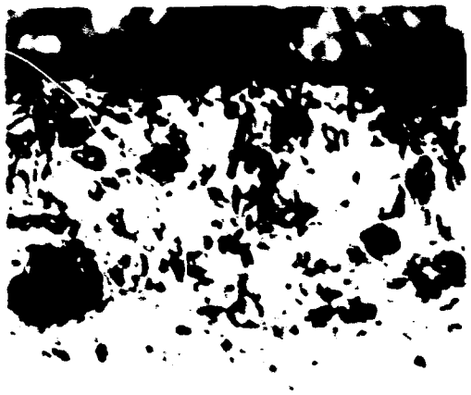

Plate 93[illegible] - .00[illegible] in.

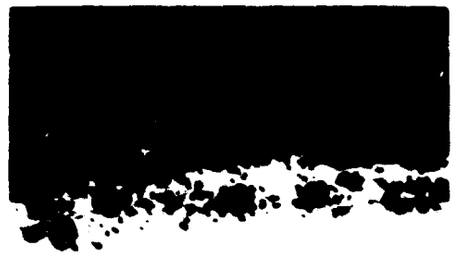

Plate [illegible] - .00[illegible] in.

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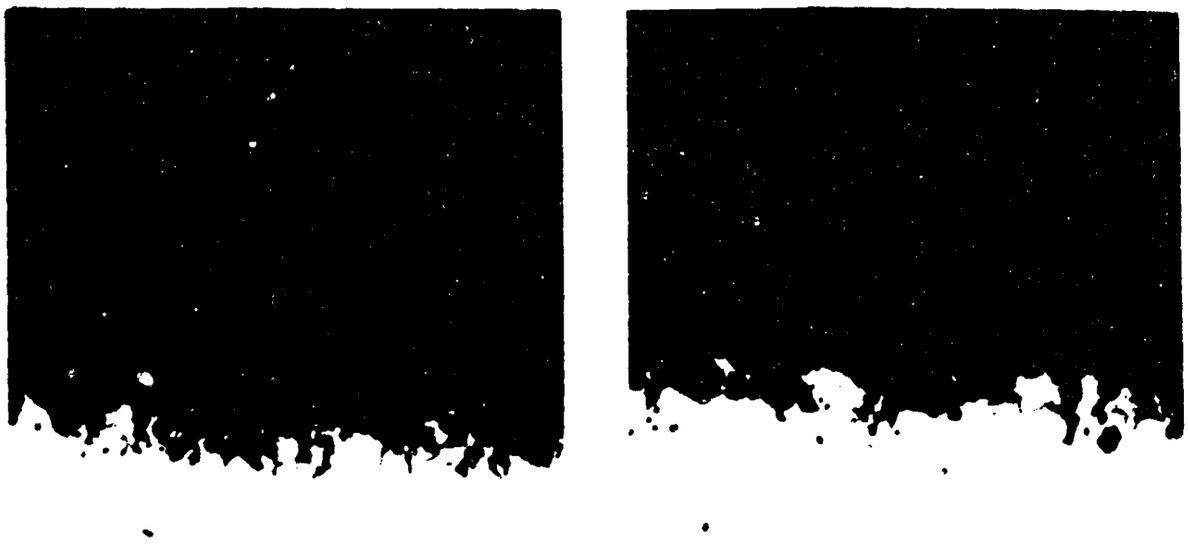
Lab. Project [illegible]
Progress Report [illegible]
Photo No. [illegible]

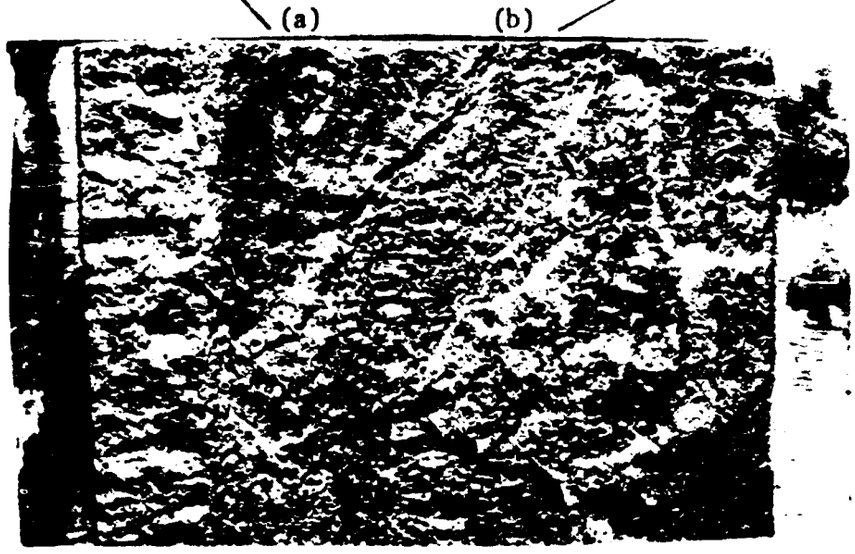
Figure 5 - Cross Sections of Mill Scale of Samples of HY-80 Plate Showing Maximum and Minimum Thicknesses (Plates S, K and 93[illegible] - 250X)



(a) Thicker Scaled Area Thickness .005 in.

(b) Thinner Scaled Area Thickness .003 in.

Magn: 250X



Magn: Approx 5X (Unpickled)

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Photo No. L-19780-6

Figure 6 - Typical Appearance of Surface of Prime HY-80 Plate and Cross Sections of Corresponding Thicker and Thinner (Spalled) Scale Areas - Plate S

TABLE 1

MILL COMPOSITIONS AND LONGITUDINAL STATIC TENSILE PROPERTIES OF 1" THICK, HY-80 PLATE

Element,%	Plate S	Plate K	Plate 93G(1)	Reg. of Spec. (2)
Carbon	.17	.14	.14	.18 Max
Manganese	.31	.34	.26	.10-.40
Phosphorus	.012	.010	.014	.025 Max
Sulfur	.007	.020	.017	.025 Max
Silicon	.27	.28	.23	.15-.35
Nickel	2.[illegible]0	2.13	2.18	2.00-3.25
Chromium	1.[illegible]0	1.39	1.13	1.00-1.80
Vandium	.003	.003	-	.03 Max
Copper	.06	.16	-	.25 Max
Molybdenum	.31	.24	.29	.20-.60
Titanium	.002	.002	-	.02 Max
Y.S., psi, 0.2% Offset	94700	89300	85600	80,000-95,000
T.S., psi	110000	107100	102700	(3)
Elong., 2",%	24.0	26.0	24.0	20.0 Min
Red. of Area, %	75.4	66.5	75.3	55.0 Min

(1) Plate was furnished under Specification MIL-S-16216C

(2) MIL-S-16216G (SHIPS) current specification

(3) To be recorded for information only

TABLE 2

SUMMARY OF PROCEDURE FOR PICKLING AND TESTING TEAR SPECIMENS

Batch Nos.	Plate Code	Pickling Time at 175°F (Hrs) (1)	Rinsing Time in Water at 175°F(min)	Tear Test Temp. °F (2)
1	S	1 1/4	2	
2	S	1 1/4	30	-80
3	S	4	2	
4	S	4	30 (3)	
1	K	1 1/4	2	-80
2	K	4	2	
1	93G	1 1/4	2	
2	93G	4	2 (4)	
3	93G	4	2	-60
4	93G	4	30	
5	93G	4	30 (4)	
1(5)	A	1	2	-90
1(5)	AA	1	2	
2(5)	AA	1	2 (6)	-90

(1) All specimens were preheated at 175°F in water prior to pickling.
(2) Prior to test, all specimens were aged at 15°F, for 24 hours, except where indicated otherwise; 15°F was used to simulate low atmospheric temperature in winter months to cover cases where plates are racked after pickling in open sheds.
(3) Rinsed for 30 min. at 200-205°F.
(4) No aging-tests made immediately after rinsing.
(5) Pickled and aged in 2" thickness, then reduced to 3/4" thickness by sawcutting to permit tear test with available capacity; maximum temperature during cutting limited to 65°F by means of coolants; time involved in cutting approximately 1/2 hour.
(6) Aged for 48 hours.

TABLE 3

PIT DEPTH MEASUREMENTS

Plate Code	Time In Pickling Bath, Hrs.	Total No. of Readings, Both Faces	Pit Depth Range, Inches		
			One Face	Other Face	Both Faces
S	1 1/4	23	.003-.032	.006-.048	.003-.048
S	2	17	.003-.054	.006-.074	.003-.074
S	4	20	.003-.053	.003-.049	.003-.053
K	1 1/4	13	.010-.031	.006-.020	.006-.031
K	2	9	.019-.026	.008-.032	.008-.032
K	4	12	.012-.031	.007-.023	.007-.031
93G	1 1/4	17	.003-.011	.003-.022	.003-.022
93G	4	15	.005-.013	.003-.034	.003-.034
S (Sandblasted)	*	16	.004-.060	.008-.051	.004-.060

* Each surface sandblasted for 1 1/2 minutes.

TABLE 4

RESULTS OF TEAR TESTS ON HY-80 STEEL, PLATE K, IN PRIME AND PICKLED CONDITIONS
(For details of pickling procedure applicable to each item, see TABLE 2)

Batch No. (1)	Pickling Time, Hrs.	Rinse Time at 175°F, Min.	Aging Time at 15°F, Hrs.	Condition	Maximum Load, Lbs./In.	Energy to Start Fracture, Ft.Lbs/In.	Energy to Propagate Fracture, Ft.Lbs./In.	Type of Fracture	% Gain or Loss Compared to Prime Plate (Avg)			
									Maximum Load	Energy to Initiate Fracture	Energy to Propagate Fracture	Total Energy
	—	—	—	Prime Plate	83,200	1230	470	Ductile				
					86,400	1170	540	Ductile				
					80,600	1140	500	Ductile				
				Average	83,400	1180	500		—	—	—	—
1	1 1/4	2	24	Pickled Plate	78,800	1020	470	Ductile				
					83,500	1180	510	Ductile				
					79,200	1010	520	Ductile				
				Average	80,500	1070	500		-3.5	-9.3	0.0	-6.5
2	4	2	24	Pickled Plate	82,000	1280	500	Ductile				
					80,700	990	560	Ductile				
					82,700	960	540	Ductile				
				Average	81,800	1080	530		-1.9	-8.5	+6.0	-4.2

(1) Tear tests performed at -80°F (1 1/2" fracture length)

TABLE 5

RESULTS OF TEAR TESTS ON HY-80 STEEL, PLATE 93C, IN PRIME AND PICKLED CONDITIONS
(For details of pickling procedure applicable to each item, see TABLE 2)

Batch No. (1)	Pickling Time, Hrs.	Rinse Time at 175°F, Min.	Aging Time at 15°F, Hrs.	Condition	Maximum Load, Lbs/in.	Energy to Start Fracture, Ft.Lbs./in.	Energy to Propagate Fracture, Ft.Lbs./in.	Type of Fracture	% Gain or Loss Compared to Prime Plate (Avg.) Maximum Load	Energy to Initiate Fracture	Energy to Propagate Fracture	Total Energy
					89,000	2160	690	Ductile (2)				
				Prime	90,000	2130	570	Ductile (2)				
	—	—	—	Plate	90,900	2050	780	Ductile				
				Average	90,300	2110	680		—	—	—	—
1	1 1/4	2	24		90,000	1830	500	Ductile (3)				
				Pickled	86,900	1700	770	Ductile				
				Plate	94,600	2030	690	Ductile (4)				
				Average	90,500	1850	650		+0.2	-12.3	- 4.4	-10.4
2	4	2	--		87,800	1650	0	Brittle				
				Pickled	86,000	1660	440	Ductile (6)				
				Plate	83,200	1490	520	Ductile (2)				
				Average	85,700	1600	320		-5.0	-24.1	-52.9	-31.1
3	4	2	24		84,600	1770	0	Brittle				
				Pickled	86,900	1840	760	Ductile				
				Plate	92,100	1860	580	Ductile (5)				
				Average	87,900	1820	450		-2.6	-13.7	-33.8	-18.6
4	4	30	24		87,700	1830	770	Ductile				
				Pickled	88,300	1950	620	Ductile (3)				
				Plate	84,600	1550	750	Ductile				
				Average	86,900	1780	710		-4.0	-15.6	+ 4.4	-10.7
5	4	30	--		92,000	2150	740	Ductile				
				Pickled	89,000	1520	740	Ductile				
				Plate	86,400	1690	760	Ductile				
				Average	89,100	1790	750		-1.3	-15.1	+10.3	- 8.9

(1) Tear tests performed at -60°F (1 1/2" fracture length)
Brittle Patches - Approx. percentage of fracture area
(2) 10%; (3) 20%; (4) 15%; (5) 20-25%; (6) 35%

TABLE 6

RESULTS OF TEAR TESTS ON HY-80 STEEL, PLATE S, IN PRIME AND PICKLED CONDITIONS
(For details of pickling procedure applicable to each item, see TABLE 2)

Batch No. (1)	Pickling Time, Hrs.	Rinse Time at 175°F, Min.	Aging Time at 15°F, Hrs.	Condition	Maximum Load, Lbs./In.	Energy to Start Fracture, Ft.Lbs./In.	Energy to Propagate Fracture, Ft.Lbs./In.	Type of Fracture	% Gain or Loss Compared to Prime Plate (Avg) Max. Load	Energy to Initiate Fracture	Energy to Propagate Fracture	Total Energy
					74,900	1190	640	Ductile				
					77,000	1240	590	Ductile				
	—	—	—	Prime	71,100	1070	580	Ductile				
				Plate	69,800	1110	510	Ductile				
					71,400	1050	510	Ductile				
					69,200	970	490	Ductile				
					76,600	1200	590	Ductile				
				Average	72,900	1120	560		—	—	—	—
1	1 1/4	2	24		70,000	1300	450	Ductile				
				Pickled	75,300	1500	490	Ductile				
				Plate	76,300	1220	520	Ductile				
				Average	73,900	1340	490		+1.3	+19.6	-12.5	+8.9
2	1 1/4	30	24		71,800	990	490	Ductile				
					76,500	1200	500	Ductile				
				Pickled	74,600	1170	490	Ductile				
				Plate	72,800	1150	480	Ductile				
					69,200	1060	470	Ductile				
					68,800	910	500	Ductile				
				Average	72,300	1080	490		-0.8	- 3.5	-12.5	-6.5
3	4	2	24		70,000	1170	450	Ductile				
				Pickled	68,800	1080	470	Ductile				
				Plate	72,800	1090	490	Ductile				
				Average	70,500	1110	470		-3.2	- 0.8	-16.0	-5.9
4	4	30(2)	--		72,700	1130	520	Ductile				
					72,500	1170	470	Ductile				
				Pickled	67,400	970	480	Ductile				
				Plate	68,900	960	440	Ductile				
					68,500	990	430	Ductile				
					70,100	1070	460	Ductile				
				Average	70,000	1050	470		-3.9	- 6.2	-16.0	-9.5

(1) Tear tests performed at -80°F (1" fracture length)
(2) Rinse temperature: 200-205°F

TABLE 7

RESULTS OF TEAR TESTS ON PICKLED 2" THICK HY-80 PLATE HEAT-TREATED TO A YIELD STRENGTH OF 100000/120000 PSI (4)
(For details of pickling procedure applicable for each item, see TABLE 2)

Batch No. (5)	Pickling Time, Hrs.	Rinse Time at 175°F, Min.	Aging Time at 15°F, Hrs.	Plate Code	Condition	Maximum Load, Lbs./In.	Ft.Lbs./In. Energy to Start Fracture	Ft.Lbs./In. Energy to Propagate Fracture	Type of Fracture	% Gain or Loss Compared to Prime Plate (Avg.) Max. Load	Energy to Initiate Fracture	Energy to Propagate Fracture	Total Energy
	—	—	—		Prime	130,400	3930	890	Ductile				
					Plate	129,400	3970	1200	Ductile				
				A	Average	128,700	4190	1570 (3)	Ductile				
						129,500	4030	1220		—	—	—	—
					Pickled	126,700	4110	1610 (3)	Ductile				
1	1	2	23 1/2	A	Plate (1)	126,700	3600	1060	Ductile				
					Average	128,200	3720	1330	Ductile				
						127,200	3810	1330		-1.8	-5.5	+9.0	-2.0
	—	—	—		Prime	120,000	3780	870	Ductile				
					Plate	121,000	3960	940	Ductile				
				AA	Average	122,000	4330	1200 (3)	Ductile				
						121,000	4020	1000		—	—	—	—
					Pickled	118,000	3530	1020	Ductile				
1	1	2	23 1/2	AA	Plate (1)	117,000	3150	960	Ductile				
					Average	118,000	3160	910	Ductile				
						117,700	3280	960		-2.7	-18.4	-4.0	-15.5
					Pickled	122,500	3620	1040	Ductile				
2	1	2	47 1/2	AA	Plate (2)	119,200	3780	920	Ductile				
					Average	120,850	3700	980		-0.1	-8.0	-2.0	- 6.7

Notes: (1) Tested 24 hours after pickling
(2) Tested 48 hours after pickling
(3) Fracture occurred at approx. 45° angle, which accounts for relatively high value
(4) Reduced to 3/4" thickness for tear tests, see note 5 in Table 2
(5) Tear tests performed at -90°F (2" fracture length)

TABLE 8

TOTAL MILL SCALE THICKNESS AND LOSS IN METAL PLATE THICKNESS DUE TO DESCALING

Plate Code	Plt. Thick; Unpickled, Avg.In. (1)	Approx.Total Mill Scale Thick.(Both Surfaces)In.	Plt. Thick After Pickling, Avg.,In. 1 1/4 Hrs.	Plt. Thick After Pickling, Avg.,In. 4 Hrs.	Plt. Thick After sand-blasting, Avg.,In.	Loss in Plate Thick; Avg., In; Exclusive of scale after Pickling (4) 1 1/4 Hrs.	Loss in Plate Thick; Avg., In; Exclusive of scale after Pickling (4) 4 Hrs.	Loss in Plt. Thick, Avg.,In., Exclussive of scale after sand-blasting (4)
S	1.034	0.007	1.024(1)	1.025 (1)	1.027 (3)	0.003	0.002	0.000
K	1.020	0.005	1.015(1)	1.015 (2)	--	0.000	0.000	---
93G	1.000	0.008	0.992(1)	0.990 (1)	--	0.000	0.002	---

(1) Each value is based on 42 determinations, 7 on each of 6 test specimens

(2) Value is based on 21 determinations, 7 on each of 3 test specimens

(3) Value is based on 13 determinations

(4) Calculated from formula indicated in paragraph 19 of report.

SND-PAPO-6302

U.S. Naval Applied Science Laboratory.
Project 9300-1.
EFFECT OF PICKLING ON NOTCH-TOUGHNESS AND SURFACE PITTING OF HY-80/100 TYPE STEEL PLATE, by F. Ginsberg, I. Geld, I.A. Schwartz and F. D'Oria. Progress Report 2. 13 Jan. 1965. 16 pp. 9 tables.
UNCLASSIFIED

Effects of acid pickling on HY-80/100 steel plate were investigated. Results indicated: (a) Pickling may be performed up to 2 hours without adverse effects provided 48 hours of aging is observed before fabrication; for shorter aging intervals, other precautions to minimize embrittlement must be observed. (b) Severe windrowing is result of mill rolling and not pickling.
Recommendations for modified pickling procedures are included.
Report concludes work on pickling of HY-80/100 steel.

1. Steel-Pickling
2. Hydrogen Embrittlement-Physical Effects
3. Ship Plates-Processing
I. Ginsberg, F.
II. Geld, I.
III. SR 007-01-01

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