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ADHESIVE BONDED STEEL: BOND DURABILITY AS RELATED TO SELECTED SURFACE TREATMENTS

A. T. DEVINE

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US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
LARGE CALIBER
WEAPON SYSTEMS LABORATORY
DOVER, NEW JERSEY

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High-strength 4340 carbon steel single lap-shear coupons were treated with four selected surface preparations: (1) wire brush, (2) sodium metasilicate/ Triton X-200 solution, (3) phosphoric acid/ethanol solution, and (4) potassium iodide/phosphoric acid (iodophosphate) solution. Bonded joints were made with all four surface preparations using 3M's EC 2214 epoxy and B.F. Goodrich's

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20. Abstract (continued)

TAME 200 acrylic adhesive. The joints were subjected to lap shear strength-control testing at 23°C and 60°C, then to hot water soak at 60°C for 100, 500, and 1,000 hours followed by testing at 60°C and stressed durability at 60°C and 95% RH until failure. Results indicate only slight differences in control test results for the different surface treatments using EC 2214, but very large differences using TAME 200. Durability of joints using both adhesives clearly favors the iodophosphate and phosphoric acid/ethanol treatments.

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INTRODUCTION

This study was designed to determine the relative durability in a harsh, warm, moist environment of bonds made to high-strength carbon steel after surface treatment by several different methods.

The selection of a surface preparation for adhesive bonding should be based upon the requirements of the system and its anticipated service environment. The combination of elevated temperature and high humidity is an adverse environment for many adhesive bonded substrates. Accelerated tests of bonded assemblies exposed to such an environment serve to identify the better surface preparations.

This work was initiated because relatively little research has been done in the area of surface pretreatments of steel, as compared with aluminum prior to bonding. This is due primarily to the fact that the aircraft related industries have provided the impetus for much of the structural bonding study done to date and aluminum, because of its favorable strength to weight ratio, is a preferred construction material for aircraft. For Army munitions applications, however, steel alloys are at least as important as aluminum alloys, and steel assemblies are increasingly being joined by adhesive bonding. The surface preparations used in this study were selected for coordination on the basis of their potential, as ascertained from the literature, and their apparent diversity in processability and chemical preparation. The wire-brush treatment used is the simplest of the surface preparations evaluated. The sodium metasilicate/Triton X-200, phosphoric acid/alcohol, and iodophosphate treatments represent recommendations of the American Society for Testing and Materials (Ref 1), a British adhesives handbook (Ref 2), and a recent American journal article (Ref 3), respectively.

DISCUSSION

Tables 1 through 3 contain the complete data necessary for a comparison of the four surface preparations. Graphical representation is shown in Figures 1 through 8.

The selection of surface preparations for bonding 4340 steel should be dictated by whether or not exposure to elevated temperatures coupled with humidity is to be encountered either in service or storage. The data in this report indicate that, with EC 2214 adhesive, any of the surface preparations are capable of yielding high-strength bonds (~35 MPa) at 23°C. At 60°C strengths are unchanged. Therefore, wire brushing appears to be a good choice on the basis of simplicity alone. If, however, the EC 2214 epoxy joints are to be exposed to elevated temperature and humidity, as simulated by the hot water soak, one may expect a steady deterioration with exposure time to the point that strengths of only 35 to 50% remain after 1,000 hours. This being the case, the use of a phosphoric acid/ethanol or iodophosphate surface preparation is advised, since these preparations yield 50% strength-retention levels; whereas, the wire brush and the wire brush followed by sodium metasilicate/Triton X-200 treatment both yielded only 35 to 40% initial strength-retention levels.

The stressed durability test, in which the EC 2214 joints were loaded to 13.70 MPa (2,000 psi) and the time-to-failure noted upon exposure to 60°C and 95% RH, indicated that the phosphoric acid/alcohol etch was most durable followed by sodium metasilicate/Triton X-200, iodophosphate, and wire brushing, in that order.

The use of TAME 200 acrylic adhesive with the same four surface treatments indicated lower initial strengths to the acid-prepared surfaces than to the wire-brushed or sodium metasilicate/Triton X-200 surfaces. Hot-water-soak tests, however, clearly favor the acid treatments since 70% to 80% of the initial strengths are retained after 1,000 hours immersion. The non-acid treatments yielded only about 40% initial-joint strengths after similar exposure. Stressed durability tests on the TAME 200 joints indicate that the acid-treated surfaces yield joints which lasted 3 to 5 times longer than the non-acid treatments.

An additional set of specimens was made using Dymax 830 epoxy adhesive over a wire-brushed steel surface coated with SAE 30 oil and dry wiped. Initial strengths were good (~30 MPa) at 23°C, but fell to ~20 MPa at 60°C. Hot-water-soak tests yielded excellent strength retention after immersion for 1,000 hours. Stressed durability test results were very poor with specimens failing in under one hour.

Surface Characteristics

Surface characterization was attempted by means of photomicrographs to determine how the topography of the surface was affected by the various methods. All surfaces were sanded prior to any subsequent treatment, so the differences in appearance are due to alterations of the surface after sanding. This alteration could have been caused by a deposition or removal of surface material.

At 6.75×10^3 magnification, the wire-brushed 4340 steel shown in Figure 9 (A) has a definite striated surface with particulate matter randomly dispersed over the surface. The striations are understandably the result of the unidirectional mechanical brushing. The particulate matter, which could have been insoluble carbon left behind after dissolving the metal from under the replica, is probably not representative of the actual wire-brushed surface and should be overlooked when interpreting this picture. Figure 10 (A) shows the wire-brushed surface after treatment with the sodium metasilicate/Triton X-200 bath. Note that the striations are still present on the surface but there is also a crusty deposit and scattered particulate matter present. In sharp contrast, the phosphoric acid/ethanol treated surface shown in Figure 11 (A) has had almost all striations masked and presents a uniformly mottled surface.

At higher magnifications such as 33×10^3 [(B), Fig 9, 10, 11] and 92×10^3 [(C), Fig 9, 10, 11] differences are even more accented. At 33×10^3 and 92×10^3 magnification, the wire-brushed surface still clearly exhibits a striated pattern, whereas the same surface post-treated with sodium metasilicate/Triton X-200 is not striated but appears blistered at these magnifications. The phosphoric acid/ethanol post-treated surface at 33×10^3 magnification is distinctly different having a glossy, almost molten or wet appearance. This points towards an amorphous or semi-crystalline surface structure. At 92×10^3 magnification, the surface possesses a sculptured look having a lightly shaded background with dark smudges creating the pattern.

New Surface Preparation

A recent Soviet journal article (Ref 4) described a process of polymerizing oils on the surface of steel to form a lacquer coating. Bonds made to these coatings, according to the Soviets, were as much as 7-1/2 times stronger than those made to degreased steel. Although all of the details of the test method were not given, the process looked promising enough to evaluate. A cursory check of the technique (using mineral oil at a temperature of 250°C for 40 minutes) resulted in bonds which were not 7-1/2 times

stronger than those formed with more commonly used surface preparations and, in fact, were only about 80% to 90% as strong. However, the results are promising enough to warrant further investigation and optimization of the technique and to check the durability of the resultant bonds under harsh temperature and humidity conditions.

Failing Modes

The predominant (~90%) mode of failure for EC 2214 RT (~23°C) control joints was adhesive for the phosphoric acid/ethanol and iodophosphate surface treatments and 50% adhesive/50% cohesive for the wire brushed and the sodium metasilicate/Triton X-200 joints. At 60°C the predominant failure mode was adhesive for all surface preparations used, although the percentage of adhesive failure for wire brushing was lower (75% adhesive/25% cohesive) than for the others.

With the TAME 200 acrylic system, all RT (~23°C) failures were adhesive in nature. At 60°C failure remained adhesive, except that for the non-acid treatments the adhesive failures were jagged (partially from each surface in a jagged pattern), whereas the acid surface preparations yielded failures which were adhesive from one side only.

After hot water soaking, the failures remained mostly adhesive (70% to 90%), but more cohesive failure was observed. Interestingly, on the wire-brushed specimens, most of the cohesive failure was located at the edge for the EC 2214 epoxy adhesive and dead center for the TAME 200 acrylic adhesive, two completely opposite failure patterns. Cohesive failure around the perimeter of a bonded joint, conditioned as these specimens were, is very unusual since water generally causes interfacial separation of the adhesive and adherend. What this unusual failing mode suggests is that a plasticizing effect is caused by the EC 2214's absorbing water into its matrix. This could weaken the adhesive sufficiently to cause it to fail cohesively before it fails adhesively.

EXPERIMENTAL

Materials

4340 Steel - An ultra high-strength steel commonly used for munitions, having the following composition: C 0.4%, Mn 0.85%, Si 0.2%, Cr 0.75%, Ni 1.80%, Mo 0.25%, and a yield strength of 270,000 psi.

EC 2214 - A one-part 120°C curing epoxy paste adhesive manufactured by the 3M Company, Minneapolis, Minnesota.

TAME 200 - A two-part room temperature (~23°C) curing acrylic adhesive manufactured by B.F. Goodrich Company, Akron, Ohio.

Surface Preparation

All 4340 steel was solvent wiped with acetone to remove excess dirt and grime. The panels were then given one of the following treatments:

Wire Brush. Steel panels were mechanically abraded with a power-driven steel wire wheel until a shiny metal appearance was achieved. The panels were then rinsed with ethanol and stored under ethanol until use.

Sodium Metasilicate/Triton X-200 (Ref 1). The steel panels were mechanically abraded as in the wire brush treatment, above, then treated according to Method E of ASTM Recommended Practice D2651. This involves immersing for 15 minutes at 63 ± 3°C (155 ± 5°F) in the following solution by weight: 47.2 parts water, 1.0 part sodium metasilicate, and 1.8 parts Triton X-200. The panels were then rinsed under running tap water and dried at 60°C.

Phosphoric Acid/Alcohol (Ref 2). The steel was abraded mechanically, as described above, then etched for 10 minutes at 60°C in the following solution by weight: 1 part orthophosphoric acid (88%) to 2 parts ethyl alcohol (denatured). The resultant smutty carbon residue was brushed off under running tap water, rinsed with de-ionized water, and dried at 120°C for 1 hour.

Iodophosphate (Ref 3). The steel panels were mechanically abraded, as described above, then alkaline cleaned in a solution of 3% (each) trisodium phosphate and sodium carbonate for 5 munites at 82 ± 3°C followed by a de-ionized water rinse. The panels were then immersed for 6 minutes in a solution of 50 g KI per ℓ of 1:1:: Conc. H_2PO_4: H_2O V/V, rinsed in de-ionized water and dried at 40°C.

Hot-Water-Soak Test

This test described in detail in PATR 4744 (Ref 5) entails immersing the bonded panels in water at 60°C and withdrawing them after 100-, 500-, and 1,000-hour immersion for lap shear tests. The shear test is carried out at 60°C immediately after removal from the water.

Stressed Durability Test

This test involves spring loading the bonded specimens to a specified percentage of its 23°C room-temperature control strength and subjecting the joint to 60°C and 95% relative humidity until failure has been induced. Load levels are generally within 20% to 40% of the control strength range, and results are plotted as load level vs time-to-failure. In this study, only the maximum (40%) load level was studied.

Single Lap Shear Test

This test, according to ASTM D1002, uses a 25.4 mm (1-in.) wide by 12.7 mm (1/2-in.) overlap joint loaded at a rate of 1.3 mm (0.05 in.) per minute until rupture. Test temperatures of 23°C and 60°C were employed.

All specimens were tested as made at 23°C and 60°C, and these data were used for controls. Subsequent tests of hot-water soaked specimens were carried out wet at 60°C.

CONCLUSIONS

There is little difference (<6%) in the strength of joints made with EC 2214 regardless of the surface preparation. After 100- and 500-hour hot water soaks there is also very little difference, but after 1,000-hour hot water soak the two phosphoric acid surface preparation methods clearly retained more strength, i.e., they yielded more durable joints.

Stressed durability results on EC 2214 joints confirm the good durability of the phosphoric acid/alcohol surface, but rate the sodium metasilicate and Triton X-200 surface more durable than the iodophosphate surface. The wire-brushed surfaces were the least durable.

Results with the TAME 200 acrylic system indicate very large differences (up to 45%) in strength among bonds made to the different surfaces, with the two phosphoric-acid prepared surfaces yielding the lowest strengths. After the 1,000-hour hot water soak, however, the joints with

the two phosphoric-acid prepared surfaces retained a substantially higher portion of their original strengths and were even higher in absolute strength, 71% and 83% for phosphoric acid/alcohol and iodophosphate, respectively, versus 37% and 40%, respectively, for the wire brushed and sodium metasilicate/Triton X-200 surfaces.

The stressed durability tests conducted on the TAME 200 specimens show clearly the outstanding durability of both of the phosphoric acid treatments employed.

Bonds approximately 60% to 300% stronger than the TAME 200 were obtained with the EC 2214 due, in part possibly, to the fact that the EC 2214 used was a single batch, one-part, 120°C curing adhesive whereas the TAME 200 was used as several batches of a two-part, room temperature (~23°C) curing system.

Both the EC 2214 and TAME 200 systems have excellent elevated temperature properties. The EC 2214 was essentially unchanged (98% to 104% of 23°C strength) at 60°C, whereas the TAME 200 was considerably stronger at 60°C (113% to 136% of 23°C strength).

Bonds to a wire brushed, thin-oil coated 4340 steel using Dymax 830 adhesive exhibited excellent hot water soak durability by retaining 100% strength after 500 hours soak and 85% after 1,000 hours soak. However, the results of the stressed durability tests were poor.

After giving 1,000-hour hot water soaked specimens an opportunity to return to original conditions (23°C + 50% RH) before testing, the TAME 200 specimens appeared to lose strength compared to those tested @ 60°C 95% RH. The EC 2214 specimens were found to gain in strength after drying out and testing @ 23°C. This indicates a tendency on the part of the EC 2214 system to recover, whereas the permanent damage to the TAME system appears much greater.

RECOMMENDATIONS

In order to more accurately determine the shape of the hot-water-soak durability curves, an additional group of data obtained at 10,000 hours (~400 days) would be most valuable. It is also strongly recommended that additional specimens be prepared and subjected to such exposure as soon as possible.

Based on some promising results obtained in bonding to a polymerized mineral-oil surface on steel, it is recommended that this steel surface preparation technique be further studied with particular attention given to the durability of the resultant joints. This technique, according to a Soviet journal article (Ref 4), has resulted in a bond 7-1/2 times stronger than that obtained on degreased steel. The process does require optimization of temperature and duration of heating for best lacquer formation from different oils.

The use of the phosphoric acid/alcohol or iodophosphate acid etch is recommended where 4340 steel surfaces are to be subjected to elevated temperature and humidity, either in service or storage. While similar trends might be expected with other steels, no extrapolation of these results to other steel alloys is advisable.

The responsiveness of other steel alloys to the surface treatments evaluated in this program should be quantitatively assessed in the same manner as the 4340 steel was assessed for this study.

The additional steps involved in using the sodium metasilicate/ Triton X-200 bath is not justified in view of the lack of improved durability observed compared with simple wire brushing. For this reason, its use is not recommended.

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Table 1

Lap shear strength and durability of TAME 200 adhesive bonded to different 4340 steel surface preparations

Surface Preparation	Control Shear Strength, MPa (psi) (23°C)	(60°C)	After Hot Water Soak @60°C, MPa (psi) (100 hr)	(500 hr)	(1,000 hr)		TTF [c] @60°C/95% RH and 4.8 MPa Stress (hr)
Wire Brushed	20.55 (2980)	25.65 (3720)	21.65 (3140)	19.37 (2810)	12.13 (1760)		506
	24.55 (3560)	25.51 (3700)	25.92 (3760)	22.89 (3320)	3.24 (470)		703
	23.10 (3350)	25.65 (3720)	25.30 (3670)	15.03 (2180)	13.17 (1910)		698
	21.58 (3130)	24.96 (3620)	21.17 (3070)	12.62 (1830)	3.59 (520)	(a)	793
	$\bar{x}$ = 22.44	$\bar{x}$ = 25.44	$\bar{x}$ = 23.51	$\bar{x}$ = 17.48	$\bar{x}$ = 9.51		$\bar{x}$ = 675
	s = 1.75	s = 0.33	s = 2.45	s = 4.56	s = 5.46		
			% Ret=92.3 [b]	% Ret=68.7	% Ret=37.3		
Sodium Meta Silicate and Triton X-200	13.79 (2000)	26.61 (3860)	22.20 (3220)	17.24 (2500)	11.91 (1730)		<837
	16.41 (2380)	24.82 (3600)	23.30 (3380)	22.20 (3220)	14.69 (2130)		96
	25.65 (3720)	23.58 (3420)	16.82 (2440)	11.72 (2180)	3.79 (550)		<837
	17.65 (2560)	24.96 (3620)	17.72 (2570)	16.75 (1830)	8.48 (1230)	(a)	37
	$\bar{x}$ = 18.37	$\bar{x}$ = 24.99	$\bar{x}$ = 20.01	$\bar{x}$ = 16.98	$\bar{x}$ = 10.13		$\bar{x}$ = <452
	s = 5.11	s = 1.25	s = 3.22	s = 4.28	s = 5.66		
			% Ret=80.1	% Ret=67.9	% Ret=40.5		
Phosphoric Acid and Alcohol	17.79 (2580)	23.03 (3340)	17.37 (2520)	11.24 (1630)	16.34 (2370)		3144
	19.31 (2800)	24.27 (3520)	16.62 (2410)	12.82 (1860)	21.79 (3160)		2496
	17.92 (2600)	25.17 (3650)	25.44 (3690)	21.30 (3090)	11.03 (1600)		2160
	15.86 (2300)	19.99 (2900)	25.99 (3770)	9.93 (1440)	9.24 (1340)	(a)	1992
	x = 17.72	x = 23.11	x = 21.36	x = 13.82	x = 16.39		$\bar{x}$ = 2448
	s = 1.42	s = 2.26	s = 5.05	s = 5.13	s = 5.38		
			% Ret=92.4	% Ret=59.8	% Ret=70.9		

Table 1 (cont)

Surface Preparation	Control Shear Strength, MPa (psi) (23°C)	(60°C)	After Hot Water Soak @60°C, MPa (psi) (100 hr)	(500 hr)	(1,000 hr)		TTF[c] @60°C/95% RH and 4.8 MPa Stress, (hr)
Iodophosphate	9.51 (1380)	11.03 (1600)	15.24 (2210)	9.24 (1340)	8.55 (1240)		1992
	11.72 (1700)	13.93 (2020)	17.37 (2520)	8.27 (1200)	17.03 (2470)		1848
	12.96 (1880)	21.10 (3060)	20.34 (2950)	16.34 (2370)	9.38 (1360)		2472
	15.51 (2250)	9.93 (1440)	13.24 (1920)	10.62 (1540)	6.76 (980)	(a)	2472
	$\bar{x}$ = 12.43	$\bar{x}$ = 14.00	$\bar{x}$ = 16.55	$\bar{x}$ = 11.12	$\bar{x}$ = 11.65		$\bar{x}$ = 2198
	s = 2.50	s = 5.03	s = 3.04	s = 3.61	s = 4.68		
			% Ret=118.2	% Ret=79.4	% Ret=83.2		

(a) Strength Retention after oven drying at 60°C for 8 hours followed by conditioning for three weeks at 23°C and 50% RH. Tested at 23°C.

(b) % Ret - Percent retention of 60°C control strength.

(c) TTF - Time to Failure

Table 2

Lap shear strength and durability of EC 2214 Adhesive bonded to several 4340 steel surface preparations

Surface Preparation	Control Shear Strength, MPa (psi) (23°C)	(60°C)	After Hot Water Soak @60°C, MPa (psi) (100 hr)	(500 hr)	(1,000 hr)	TTF[c] @60°C/95% RH and 13.8 MPa Stress, (hr)
Wire Brushed	35.51 (5150)	33.78 (4900)	25.99 (3770)	26.13 (3790)	15.65 (2270)	116
	35.99 (5220)	35.85 (5200)	25.58 (3710)	25.51 (3700)	12.82 (1860)	122
	34.61 (5020)	36.40 (5280)	23.03 (3340)	24.61 (3570)	14.69 (2130)	111
	36.13 (5240)	34.34 (4980)	27.58 (4000)	22.75 (3300)	26.48 (3840)[a]	98
	$\bar{x}$ = 35.56	$\bar{x}$ = 35.09	$\bar{x}$ = 25.55	$\bar{x}$ = 24.75	$\bar{x}$ = 14.39	$\bar{x}$ = 112
	s = .69	s = 1.24	s = 1.89	s = 1.47	s = 1.44	
			% Ret=72.8[b]	% Ret =70.5	% Ret =41.0	
Sodium Meta Silicate and Triton X-200	34.61 (5020)	38.33 (5560)	26.34 (3820)	9.93 (2890)	7.72 (1120)	150
	36.54 (5300)	39.16 (5680)	27.65 (4010)	26.54 (3850)	15.31 (2220)	132
	40.54 (5880)	--	29.37 (4260)	26.96 (3910)	17.24 (2500)	179
	37.09 (5380)	38.89 (5640)	29.30 (4250)	26.89 (3900)	27.37 (3970)[a]	188
	$\bar{x}$ = 37.20	$\bar{x}$ = 38.79	$\bar{x}$ = 28.17	$\bar{x}$ = 25.08	$\bar{x}$ = 13.42	$\bar{x}$ = 162
	s = 2.47	s = 0.42	s = 1.46	s = 3.44	s = 5.03	
			% Ret=72.6	% Ret=64.7	% Ret=34.6	
Phosphoric Acid and Alcohol	39.16 (5680)	38.33 (5560)	24.48 (4130)	25.72 (3730)	19.24 (2790)	201
	34.68 (5030)	39.30 (5700)	27.79 (4030)	21.44 (3110)	19.86 (2880)	209
	34.34 (4980)	35.44 (5140)	28.06 (4070)	24.82 (3600)	20.13 (2920)	195
	39.30 (5700)	34.13 (4950)	27.03 (3920)	24.68 (3580)	24.82 (3600)[a]	262
	$\bar{x}$ = 36.87	$\bar{x}$ = 36.80	$\bar{x}$ = 27.84	$\bar{x}$ = 24.17	$\bar{x}$ = 19.74	$\bar{x}$ = 217
	s = 2.73	s = 2.42	s = 0.61	s = 1.87	s = 0.46	
			% Ret=75.7	% Ret=65.7	% Ret=53.6	

Table 2 (continued)

Surface Preparation	Control Shear Strength, MPa (psi) (23°C)	(60°C)	After Hot Water Soak @60°C, MPa (psi) (100 hr)	(500 hr)	(1,000 hr)	TTF[c] @60°C/95% RH and 13.8 MPa Stress, (hr)
Iodophosphate	39.16 (5680)	39.99 (5800)	26.96 (3910)	28.27 (4100)	19.65 (2850)	116
	35.16 (5100)	36.96 (5360)	26.82 (3890)	29.30 (4250)	20.55 (2980)	136
	37.58 (5450)	37.78 (5480)	26.96 (3910)	24.13 (3500)	18.89 (2860)	169
	38.61 (5600)	37.65 (5460)	27.44 (3980)	27.79 (4030)	26.89 (3900) [a]	171
	$\bar{x}$ = 37.63	$\bar{x}$ = 38.09	$\bar{x}$ = 27.04	$\bar{x}$ = 27.37	$\bar{x}$ = 19.70	$\bar{x}$ = 148
	s = 1.77	s = 1.31	s = 0.27	s = 2.25	s = 0.83	
			% Ret=71.0	% Ret=71.9	% Ret=51.7	

(a) Strength retention after oven drying at 60°C for 8 hours followed by conditioning for three weeks at 23°C and 50% RH. Tested at 23°C.

(b) % Ret - Percent retention of 60°C control strength.

(c) TTF - Time to Failure

Table 3

Lap shear strength and durability of Dymax 830 Adhesive bonded to wire brushed/oil coated 4340 steel

Surface Preparation	Control Shear Strength, MPa (psi) (23°)	(60°)	After Hot Water Soak @60°C, MPa (psi) (100 hr)	(500 hr)	(1,000 hr)	TTF(b) @60°C/95% RH and 12.4 MPa Stress, (hr)
Wire Brushed then Oil Coated	31.03 (4500)	19.72 (2860)	21.51 (3120)	14.07 (2040)	11.31 (1640)	<1
	31.72 (4600)	17.65 (2560)	28.06 (4070)	22.75 (3300)	22.06 (3200)	<1
	28.96 (4200)	25.65 (3720)	21.93 (3180)	21.99 (3190)	18.55 (2690)	<1
	32.54 (4720)	18.62 (2700)	23.37 (3390)	22.82 (3310)	17.51 (2540)	<1
	x = 31.06	x = 20.41	x - 23.72	x = 20.41	x - 17.36	$\bar{x}$ = <1
	s = 1.53	s = 3.59	s - 3.00	s = 4.25	s - 4.48	
			% Ret=116.2(a)	% Ret=100	% Ret=85.1	

(a) % Ret - Percent retention of 60°C control strength.

(b) TTF - Time to Failure

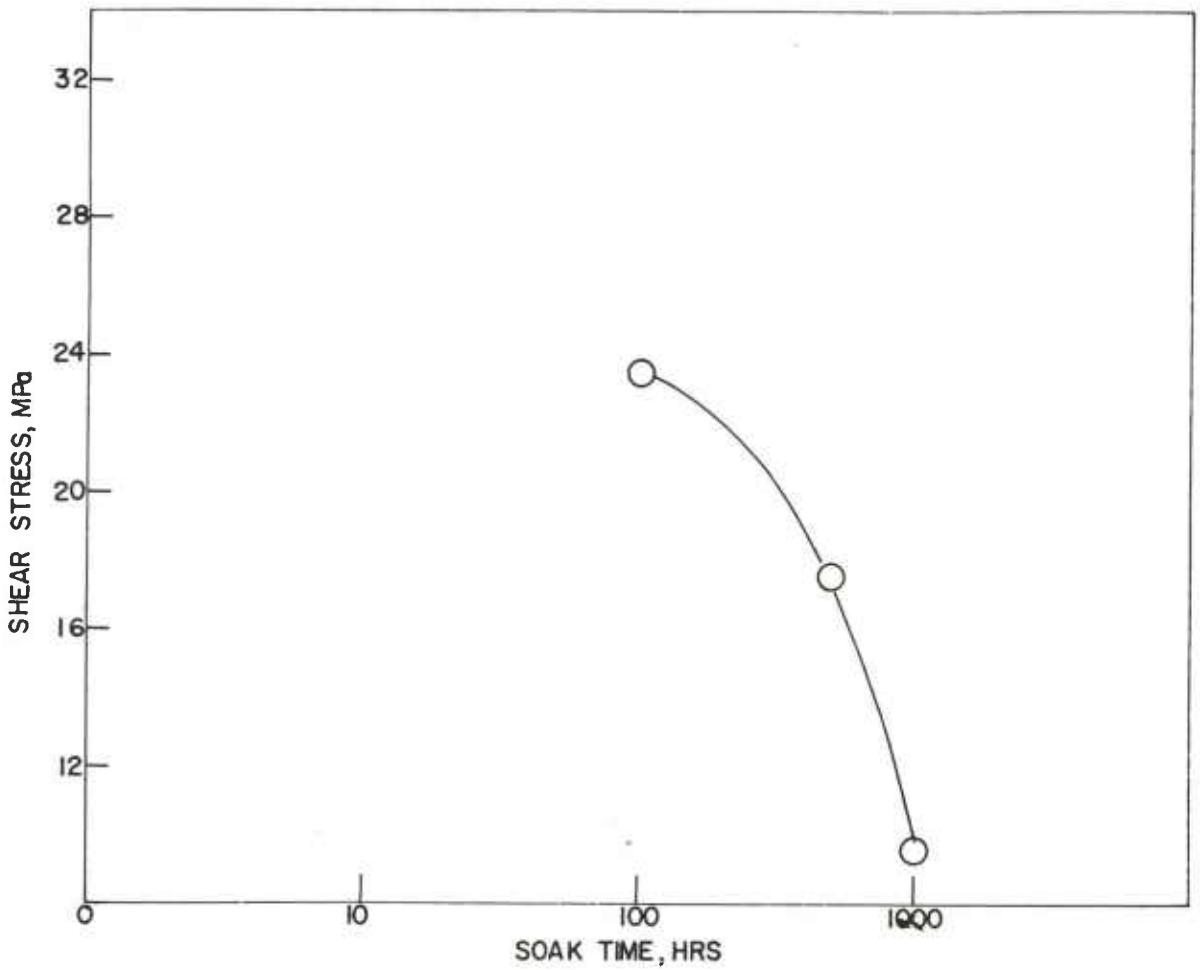


Figure 1. TAME 220/wire brushed steel, residual shear strength after 60°C hot water soak

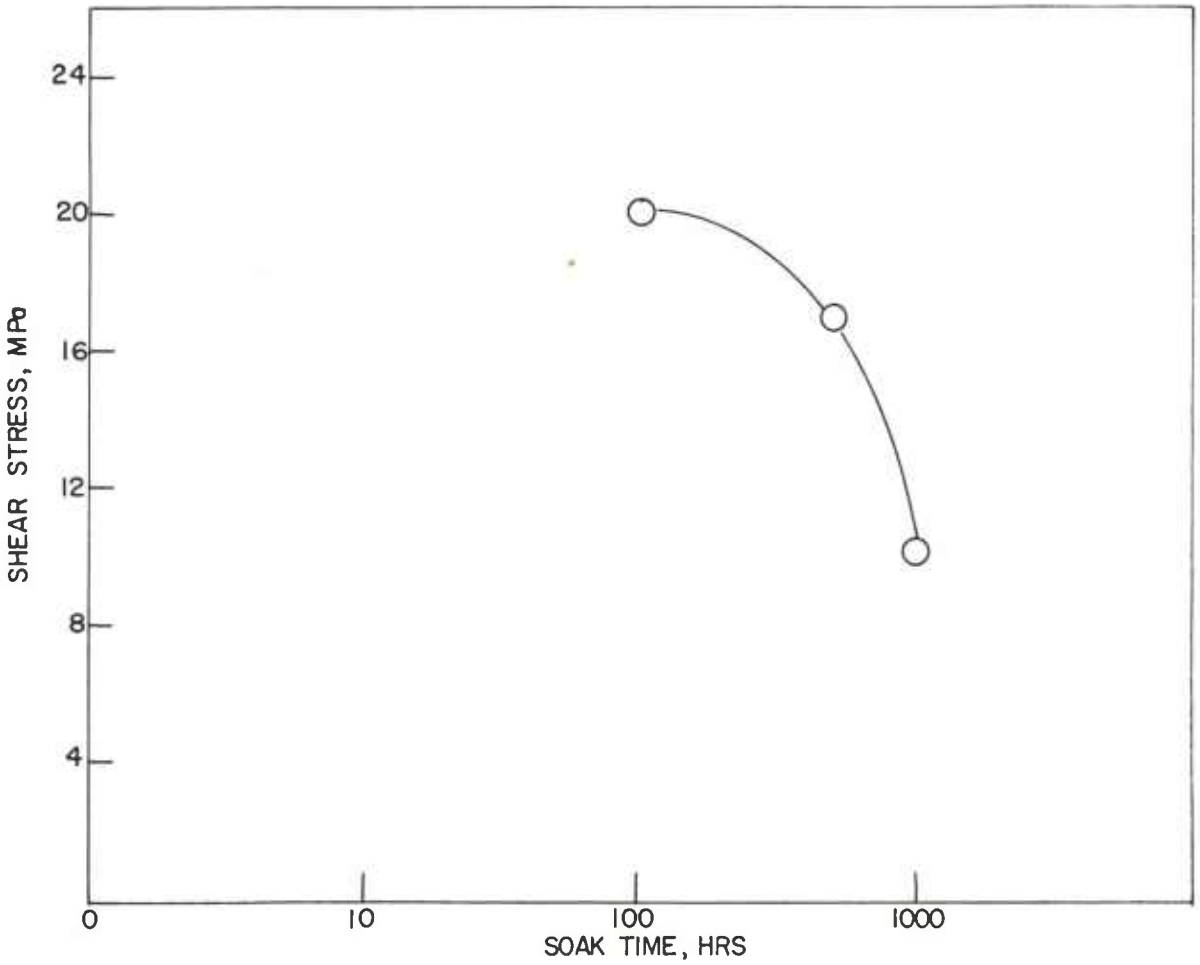


Figure 2. TAME 200/sodium metasilicate + TX 200, residual shear stress after 60°C hot water soak

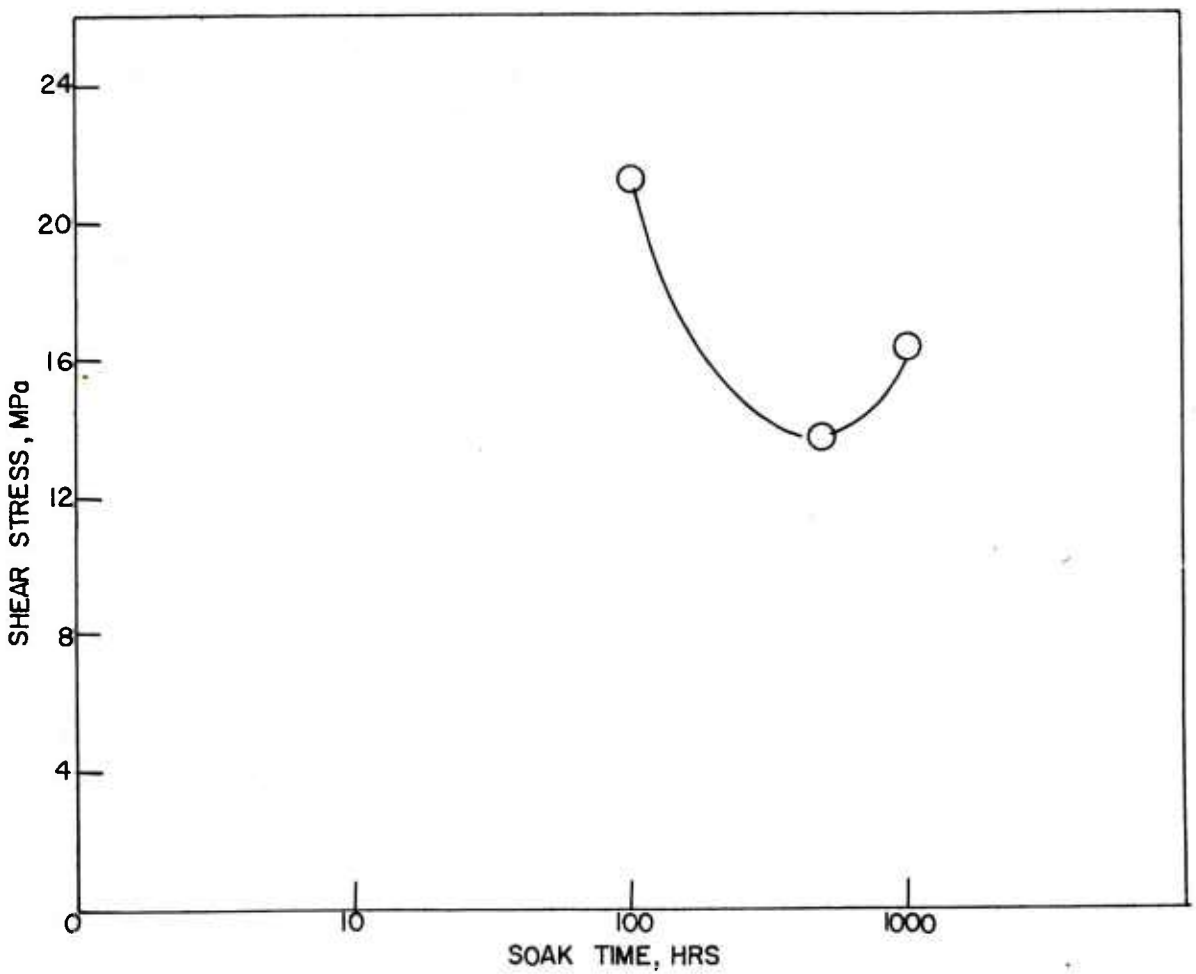


Figure 3. TAME 200/phosphoric acid-alcohol, residual shear strength after 60°C hot water soak

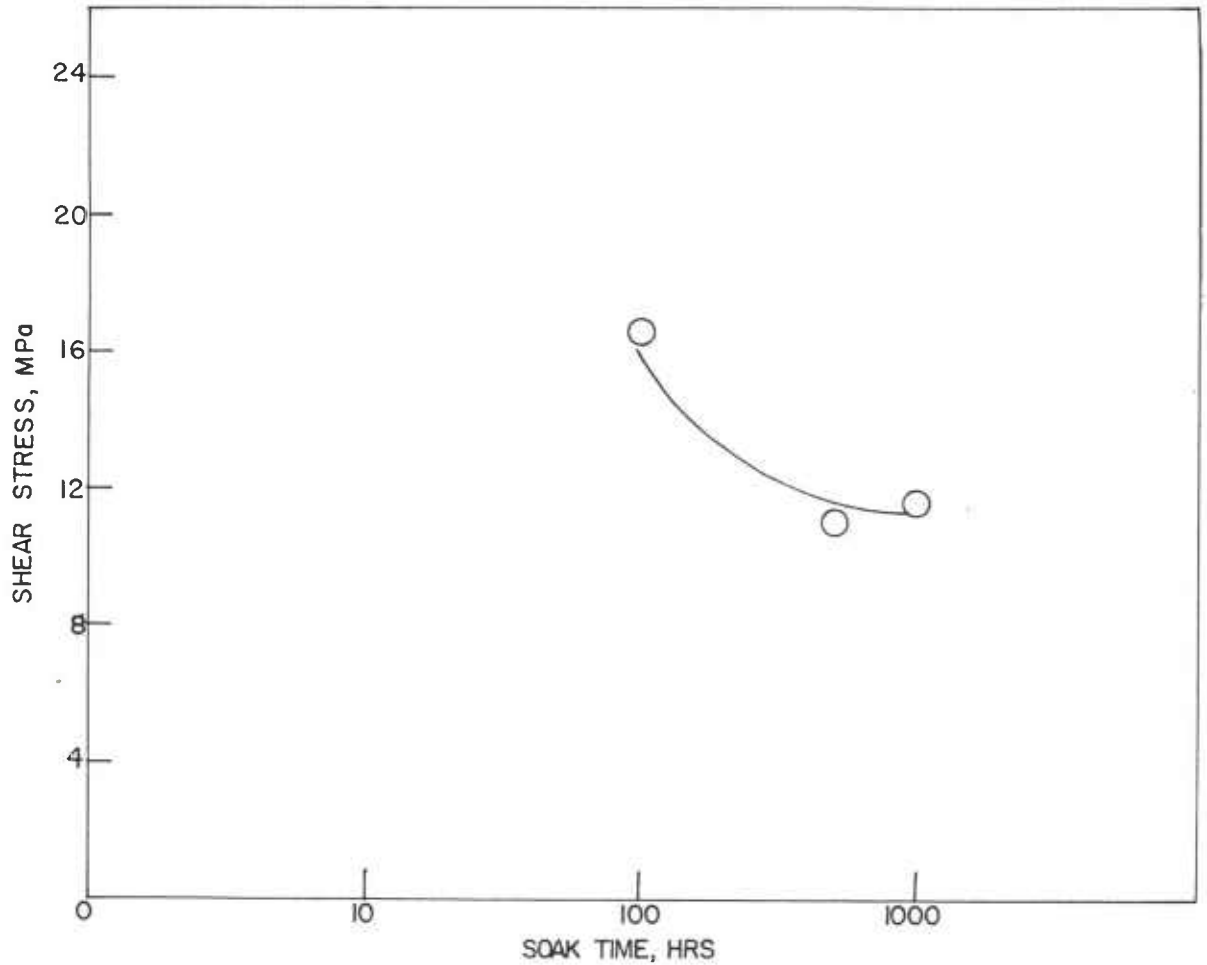


Figure 4. TAME 200/iodophosphate, residual shear strength after 60°C hot water soak

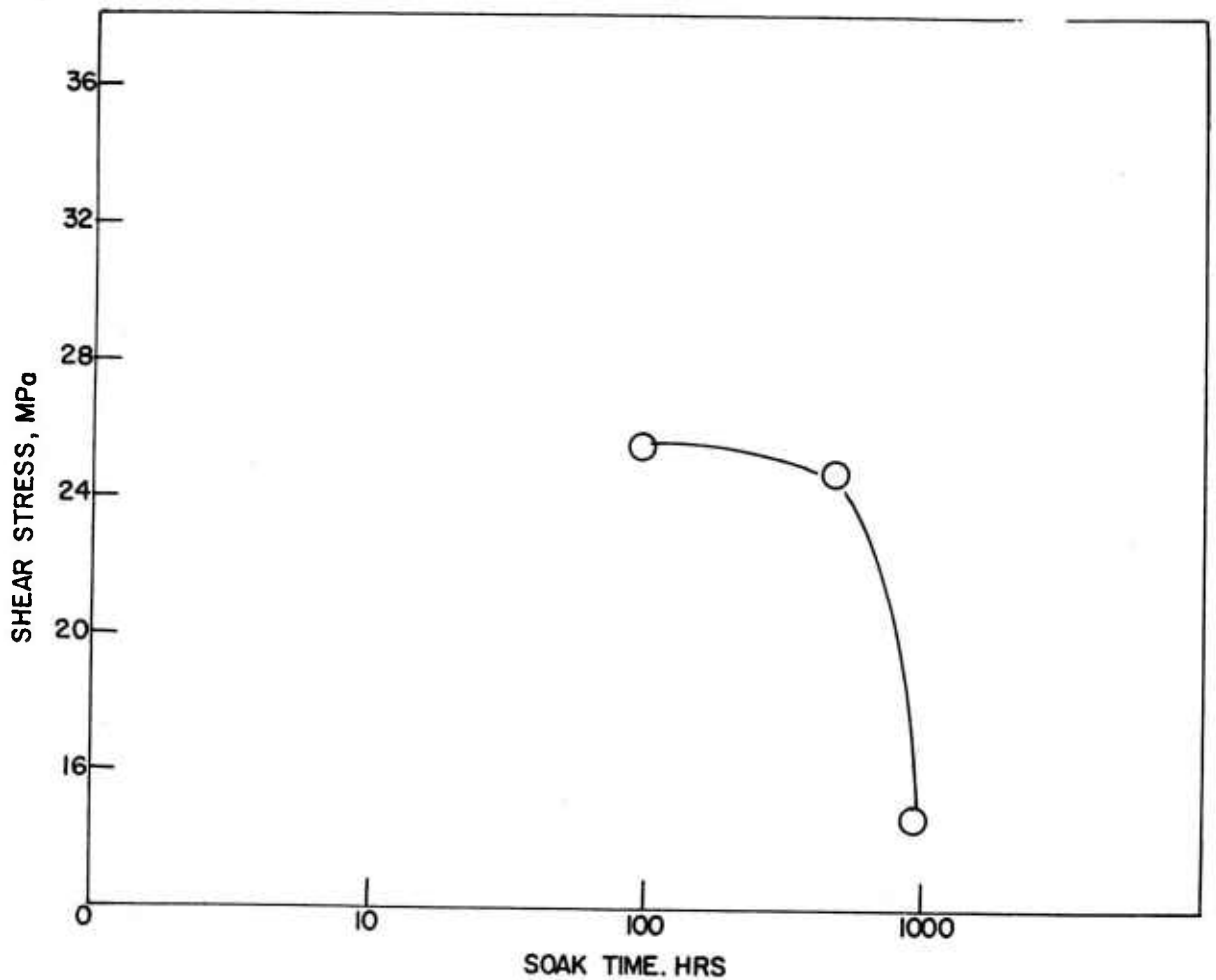


Figure 5. EC 2214/wire brushed, residual shear strength after 60°C hot water soak

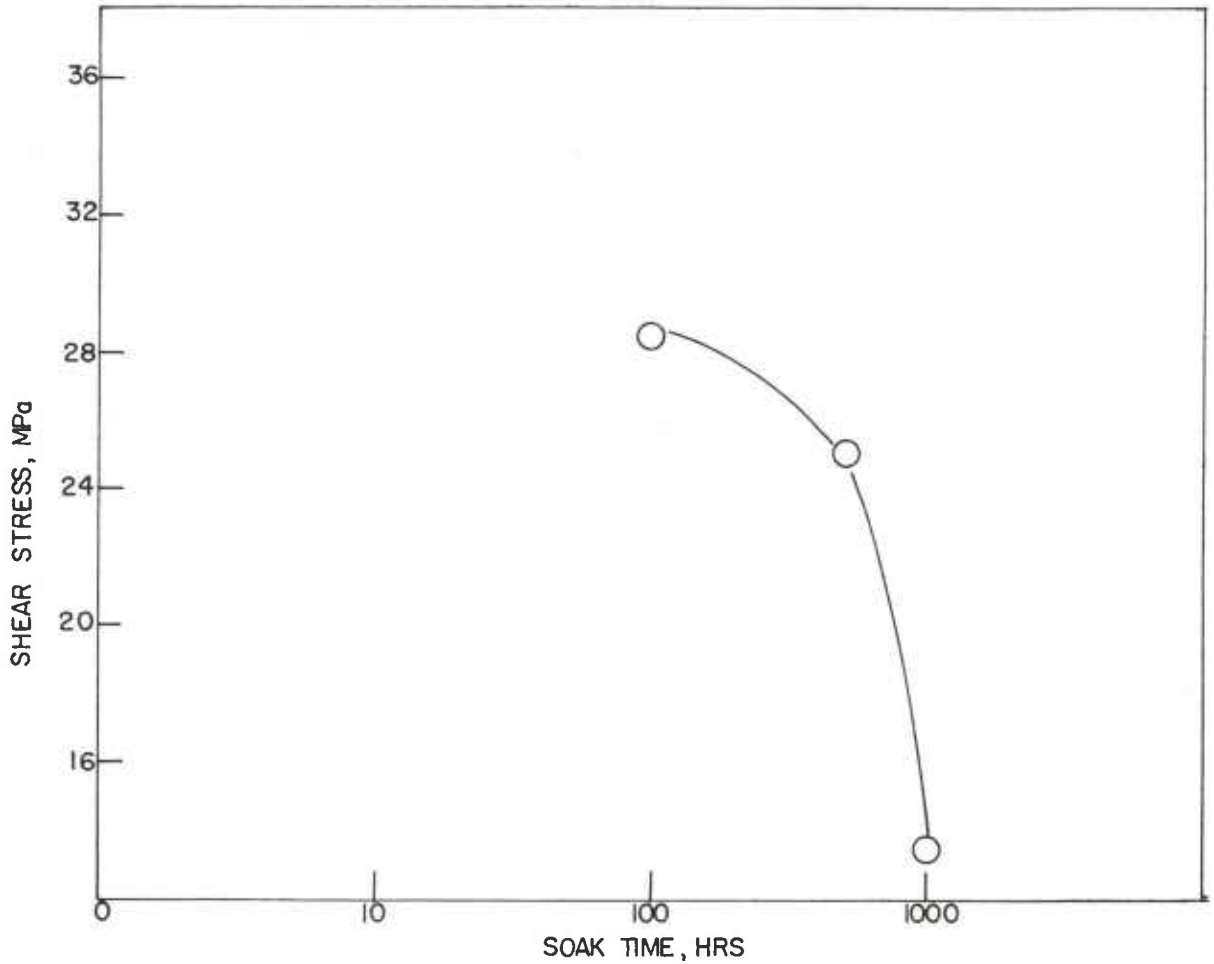


Figure 6. EC 2214/sodium metasilicate + TX 200, residual shear strength after 60°C hot water soak

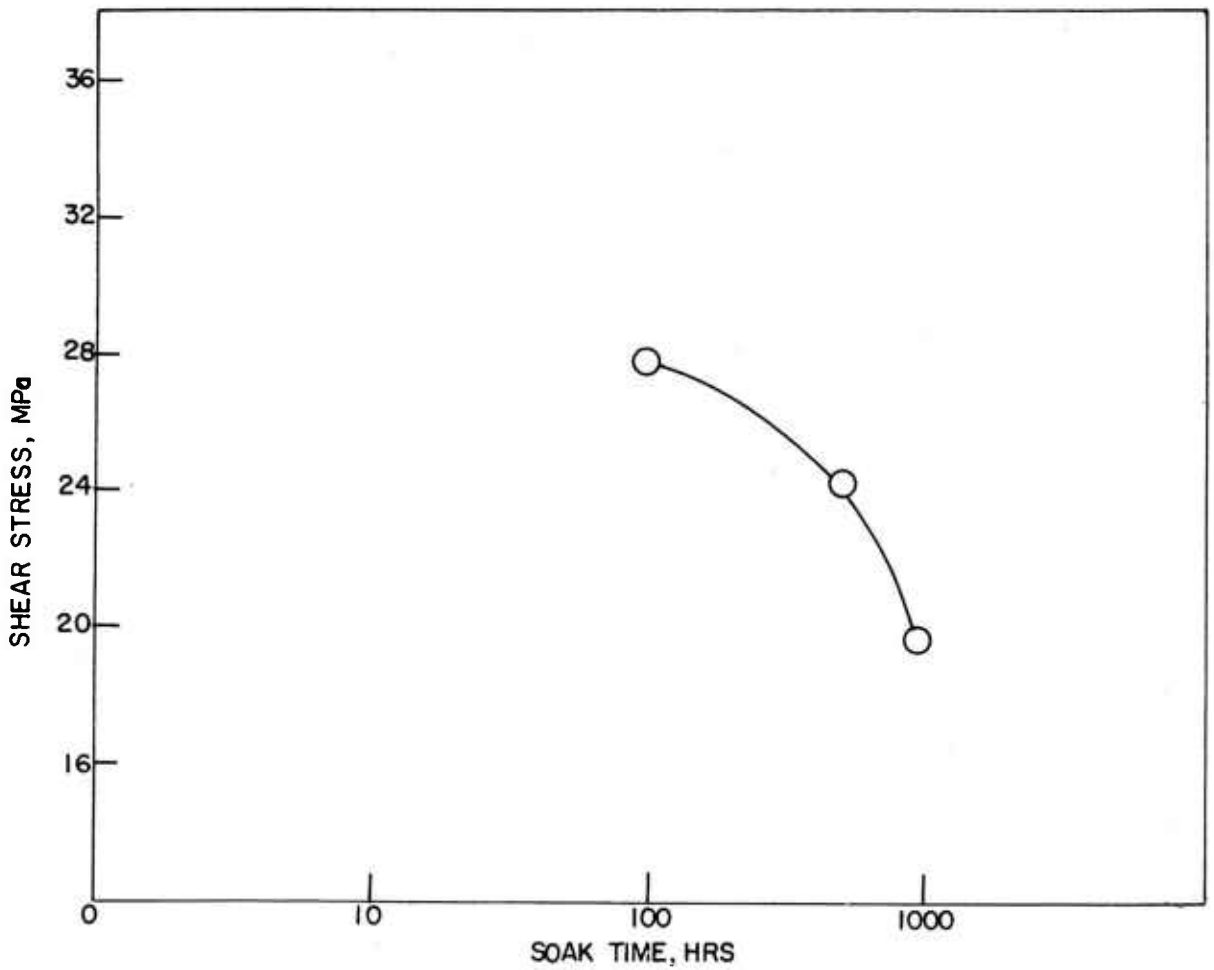


Figure 7. EC 2214/phosphoric acid + alcohol, residual shear strength after 60°C hot water soak

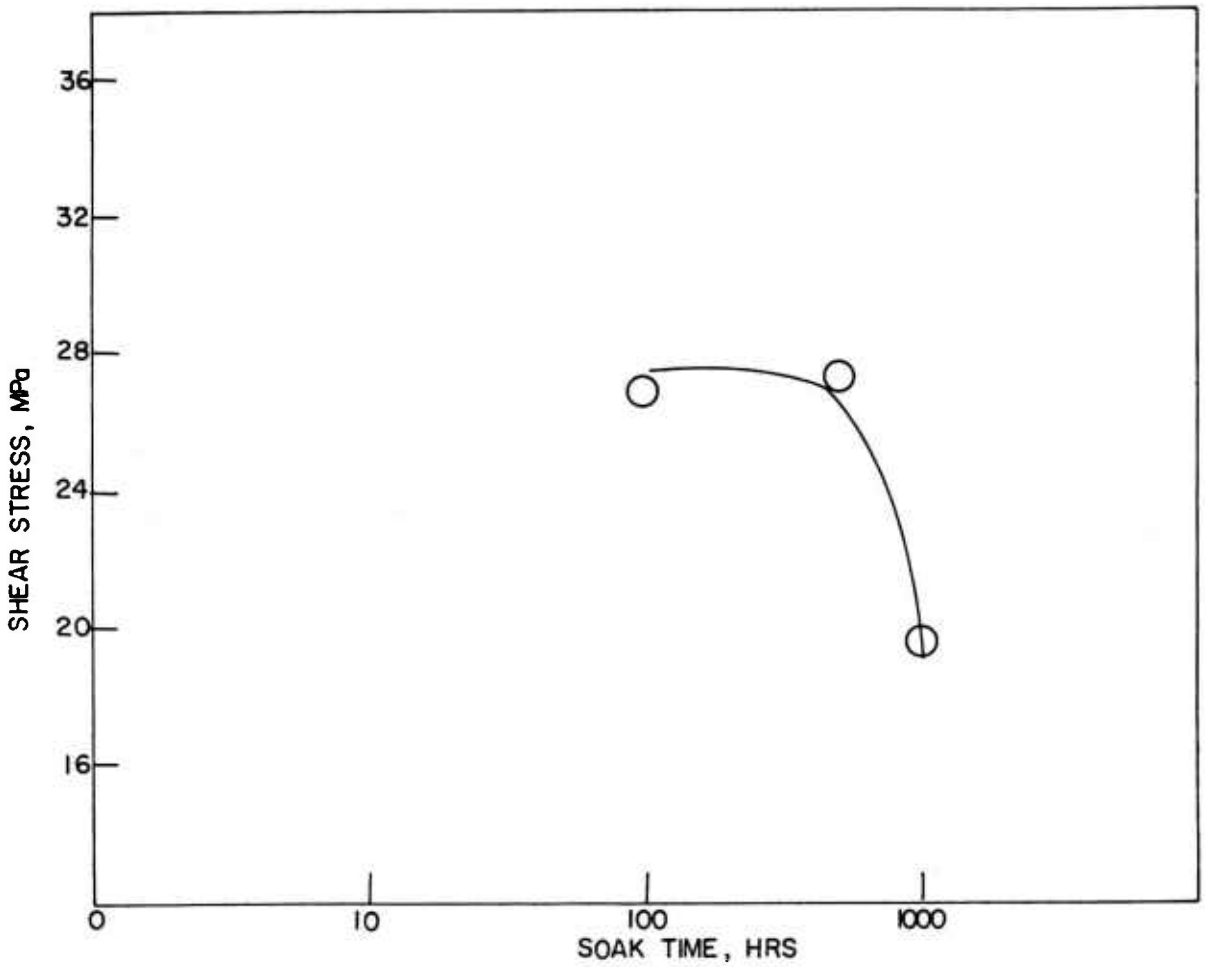


Figure 8. EC 2214/iodophosphate, residual shear strength after 60°C hot water soak

(A) 6,750 X

(B) 33,000 X

Figure 9. Wire brushed 4340 steel

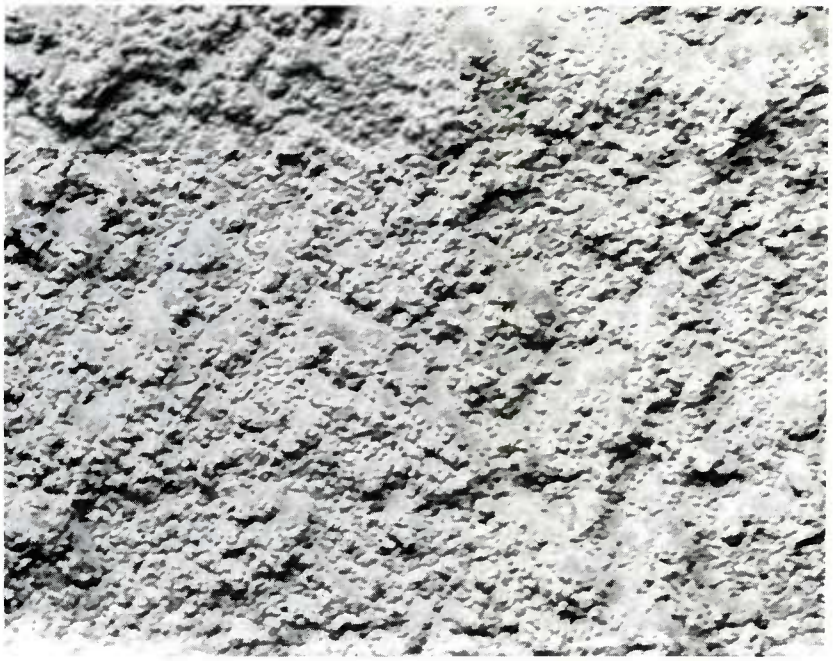
(C) 92,000 X

Figure 9. Wire brushed 4340 steel (continued)

(A) 6,750 X

(B) 33,000 X

Figure 10. Wire brushed 4340 steel with sodium metasilicate/Triton X-200 post treatment



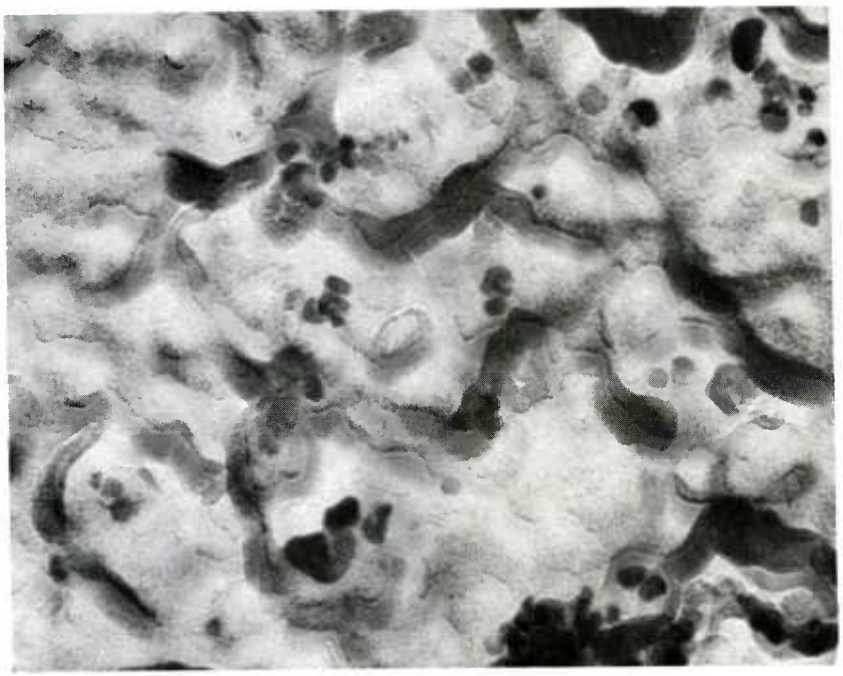
(C) 92,000 X

Figure 10. Wire brushed 4340 steel with sodium metasilicate/Triton X-200 post treatment (continued)

(A) 6,750 X

(B) 33,000 X

Figure 11. Wire brushed 4340 steel with phosphoric acid/ethanol post treatment



(C) 92,000 X

Figure 11. Wire brushed 4340 steel with phosphoric acid/ethanol post treatment (continued)

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