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PRELIMINARY RESULTS OF A COMPARATIVE STUDY OF SEVERAL COMMERCIALLY AVAILABLE OXIDATION RESISTANT COATINGS ON Mo—0.5 Ti. ALLOY SHEET

by

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PRELIMINARY RESULTS OF A COMPARATIVE STUDY OF SEVERAL COMMERCIALLY AVAILABLE OXIDATION RESISTANT COATINGS ON Mo–0.5 Ti ALLOY SHEET

The Langley Research Center of the NASA is currently investigating various commercially available protective coatings for refractory metals and their alloys. This program is concerned primarily with coatings for thin sheet which will have structural applications above 2000° F. The current program includes continuous and cyclic oxidation tests in static air, flowing air and reduced pressure static air oxidation tests, and room temperature mechanical property tests. The effects of various substrate edge conditions on coating life is also being investigated. In addition, metallurgical examinations are being made on the coatings before and after high temperature exposure. The data presented here are the preliminary results of oxidation tests in static air at 2500° F and include constant and cyclic temperature tests, tests on three edge conditions, and room temperature tensile tests on coated specimens.

Specimens

The specimens for this program are shown in figure 1. Included are oxidation tabs, tensile specimens, and leading edge specimens. All specimens were fabricated from stress-relieved Mo–0.5 Ti sheet 0.012 in. thick. Several oxidation tabs were made with each of the following edge conditions:

1. Warm sheared to size.
2. Machined to size and edges hand broken with emery.
3. Machined to size with 0.125" radii at corners. All edges were then dry tumbled in an abrasive mix.

The specimens were all fabricated by NASA and forwarded to various coating suppliers for the application of their best available coating.

All specimens were prepared from the same material and all suppliers received the same coating specifications.

The specifications furnished guidelines for the coating which would represent the best compromise between maximum oxidation resistance and minimum alteration of the uncoated mechanical properties of the substrate. The suggested guidelines included the following:

1. Oxidation resistance of at least 10 hours at 2500° F in static air.
2. A maximum allowable decrease in room temperature elongation of 25%.
3. A maximum allowable weight increase of 25%.

In addition to the performance guides, the coating supplier was requested to apply the same coating to all specimens and to use his normal cleaning and surface preparation procedures prior to coating. A list of the coating suppliers for this program is presented in Table I which also includes the coating designation, coating thickness, substrate thickness (before and after coating), and the weight gain of the coated specimen. The weight gain for the coated specimens includes the effects of any surface removal of the substrate by the coating supplier, prior to coating. The Disil specimens, for instance, showed a net weight loss, after coating.

Equipment

Constant temperature tests. - Static air, continuous exposure, testing is being conducted in a vertical tube furnace, a schematic view of which is shown in figure 2. The oxidation tabs are supported in a zircon boat which is suspended on a platinum wire in the furnace. The automatic continuous recording balance is used to record weight changes versus time. The balances presently in use are capable of detecting a specimen weight change of 0.2%, and continuously record a weight change up to 20% for the oxidation tabs in this study. The cyclic testing is performed in a horizontal box furnace. A small hole has been cut in the furnace door for specimen insertion, and a zircon boat supports the specimens.

Testing Procedures. - The types of oxidation tests used in this program are indicated in figure 3. The constant temperature tests expose the specimen continuously to the test temperature until failure. A 10% weight loss was used as the failure criterion. If failure was not observed after 250 hours, the test was normally terminated.

In two of the cyclic tests the specimen was inserted into the furnace with 95% of the temperature change being achieved in 30 seconds. At the completion of the cycle the specimen was removed rapidly from the furnace and allowed to air cool.

The other cyclic test controls a slow linear temperature rise rate for 1/2 hour, exposes the specimen to the test temperature for 1 hour, and then controls cooling rate for 1/2 hour.

In the cyclic tests the specimens are weighed after each cycle. A weight loss of at least 10% terminates the cyclic tests.

RESULTS

Constant Temperature Oxidation Tests. - Typical plots of weight change versus time for the various coatings are shown in figure 4. Some coatings may lose as much as 3% of their original weight during the first hour. This loss of weight is usually accompanied by smoking, which gradually stops as the coating heals.

The specimen may then remain at the same weight for many hours before catastropic failure occurs. In view of this mode of failure, the evolution of smoke or a small weight change as a criteria for failure may not result in a realistic assessment of the protection provided by the coating. It appears, however, that after 5 to 6% weight loss the various coatings are all failing rapidly with the possible exception of the two AMF coatings.

Cyclic Oxidation Tests. - Accumulated times at 2500° F before failure for the various coatings under cyclic and continuous exposure oxidation tests are compared in figure 5. The figure clearly indicates the severity of the cyclic tests for the majority of the coatings. As expected, the coatings with the longest constant temperature life also exhibited superior cyclic exposure life. In terms of accumulated time, the 0.1 hour cycle was the most severe cyclic test. In terms of the number of cycles, a coating would generally withstand as many or more 0.1 hour cycles than 1.0 hour cycles before failure.

Edge Effects. Edge preparation is generally considered to be an important factor in coating life. Photomicrographs (figure 6) for one of the coatings before exposure are typical of the as-coated appearance for the three edge conditions under study. As expected, the coating on the tumbled edge appears superior to the other two. The partially recrystallized substrate of the tumbled edge and the sheared edge would suggest that these two edge preparations are less severe than the fully recrystallized substrate of the machined edge. The lower recrystallization temperature of the broken edge, which may be the result of a more severe fabrication process and the accompanying higher residual stresses, could contribute to a shorter life for a coating applied to this edge condition. Comparison of the lifetimes for these various edge conditions (figure 7) suggests that the edge effect is quite pronounced. With the exception of the CV and AMF coatings both the sheared and tumbled edges provided longer lifetimes than the broken edges.

Tensile Tests. – Room temperature tensile tests were performed on the coated tensile specimens in the as-coated condition and compared with control test data for the base metal. Figure 8 shows tensile strength, 0.2% offset yield strength, and elongation results for the coated specimens compared as a ratio to the base metal properties.

In general, the coated specimens show a decrease in mechanical properties. However, the reductions in some cases of yield and tensile strengths are in excess of those to be expected by the reduction in substrate area (table 1). In general, the thicker coatings which had the longest lifetimes also produced the more severe reductions in the mechanical properties of the coated tensile specimens (figure 8).

CONCLUSIONS

From the preliminary results presented herein, the following may be concluded.

1. Thicker coatings generally give longer lifetimes in both static and cyclic tests but have the most severe effect on the mechanical properties of the substrate.

2. A failure criterion of about 6 percent weight loss appears reasonable for the specimens tested. Other failure criteria such as evolution of smoke or smaller weight losses may not give realistic data.

3. Cyclic oxidation testing is much more severe on all coatings tested then continuous static tests, in terms of lifetimes.

4. Sheared edges and tumbled edges on the substrate before coating generally provide longer lifetimes than machined and broken edges.

ADDITIONAL TESTING

Additional testing is planned to expand the present data and include other test temperatures. The effects of flowing air and static air at reduced pressure on the life of the coatings under study will also be investigated.

TABLE 1. – COATINGS UNDER STUDY

Supplier	Designation	Avg. Coating Thickness (in.) [a]	Substrate Thickness (in.) [a]		Wt. Change (percent)
			Before Coating	After Coating	
American Machine & Foundry	AMF (Glassed) [b]	0.0017	0.0122	0.0092	2.20
American Machine & Foundry	AMF (Not Glassed)	0.0017	0.0122	0.0099	2.20
Boeing	Disil	0.0015	0.0108	0.0081	-3.81
Chance Vought	C-V	0.0014	0.0120	0.0106	3.43
Chromalloy	W-2	0.0012	0.0121	0.0109	4.40
Pfaudler	PFR-5	0.0023	0.0118	0.0091	13.67
Pfaudler	PFR-6	0.0019	0.0116	0.0097	7.92

NOTE: (a) Based on cross section of tumbled specimen

(b) Preglassed 1 hour at 2800° F by AMF

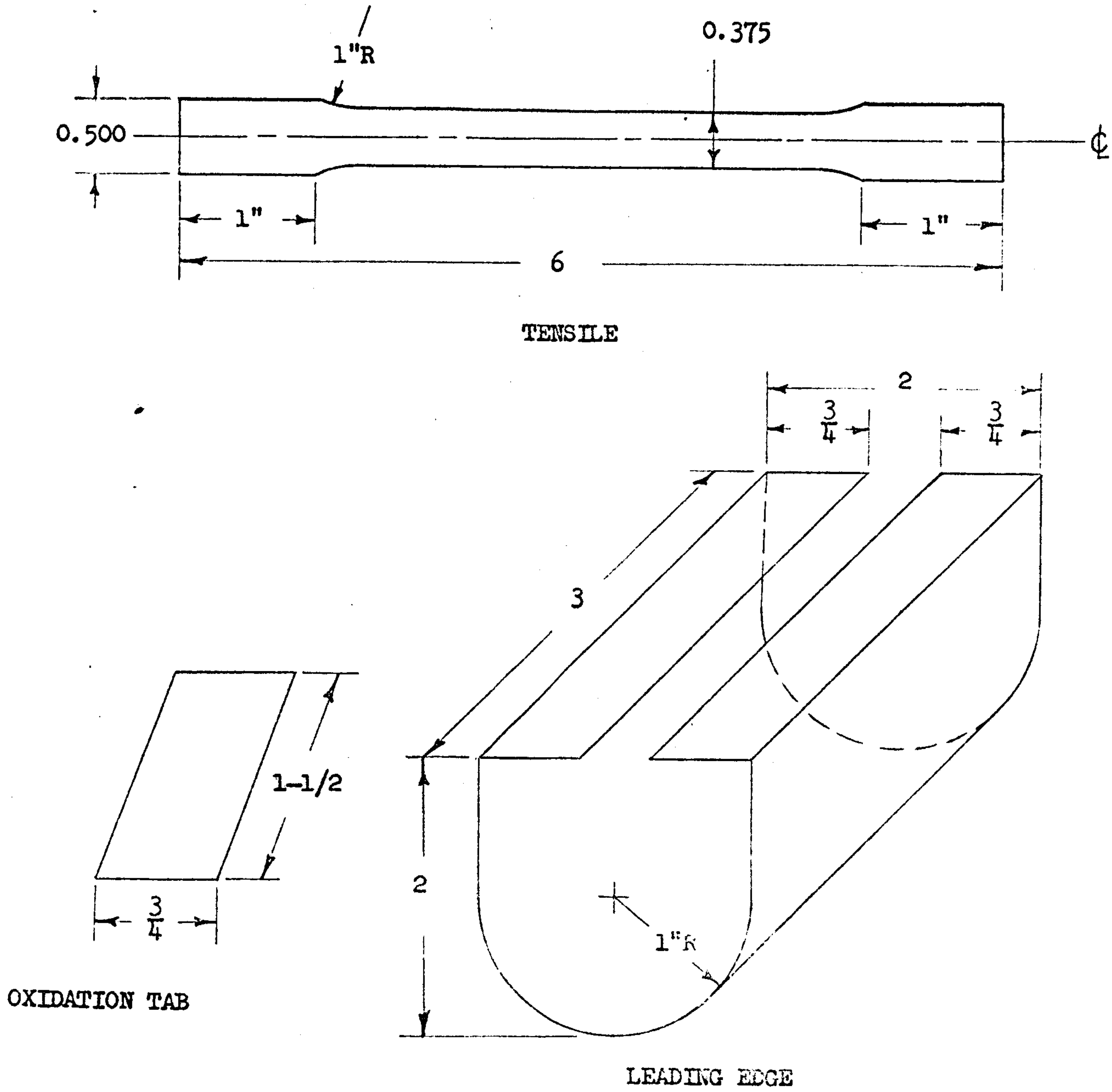

Figure 1. – SPECIMEN CONFIGURATIONS FOR COATING INVESTIGATIONS

Note: Spec. thickness 0.012" before coating.

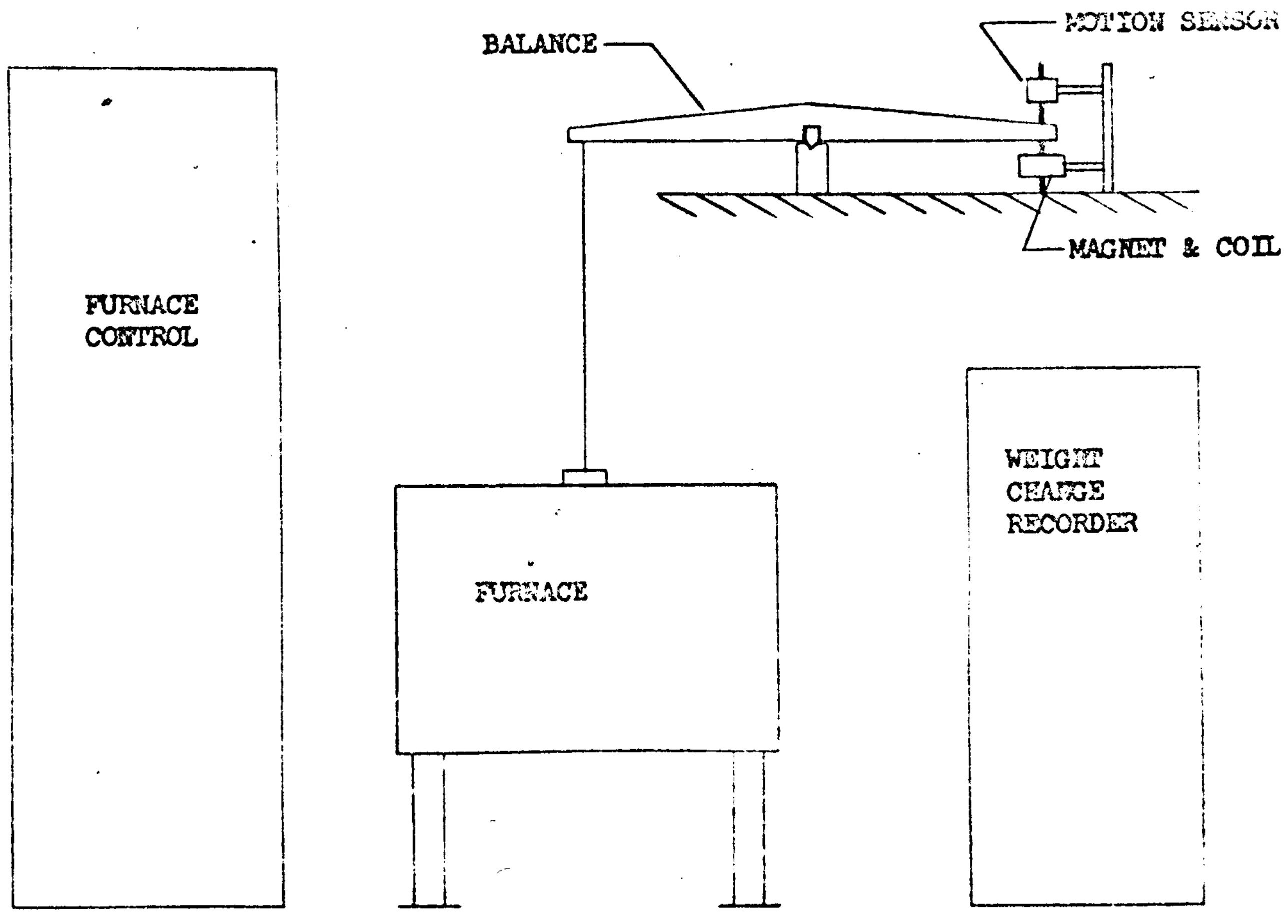

Figure 2. - EQUIPMENT FOR CONSTANT TEMPERATURE STATIC AIR OXIDATION TESTS.

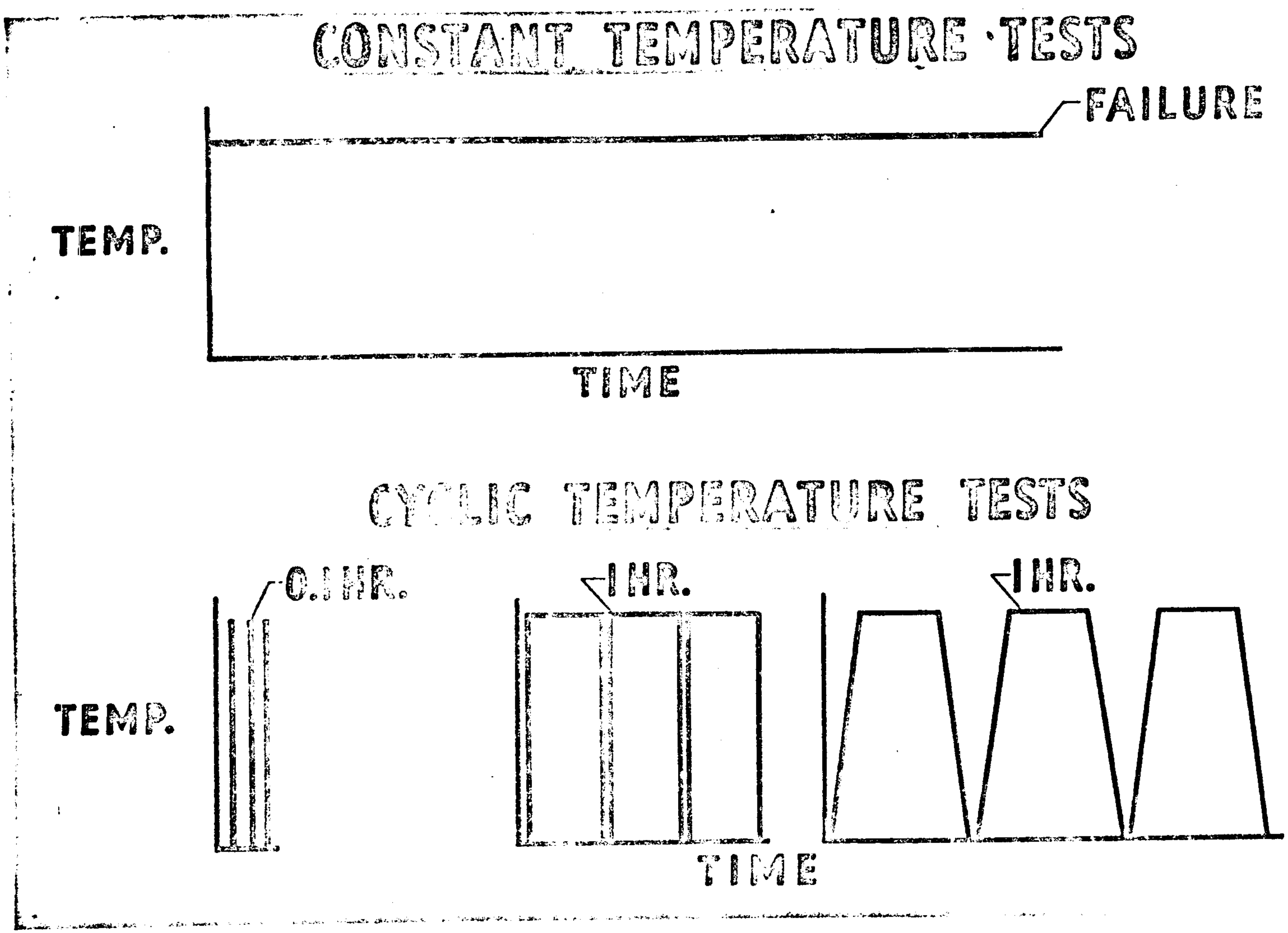

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Figure 3. – OXIDATION TESTS UNDER CONSTANT OR CYCLIC TEMPERATURES

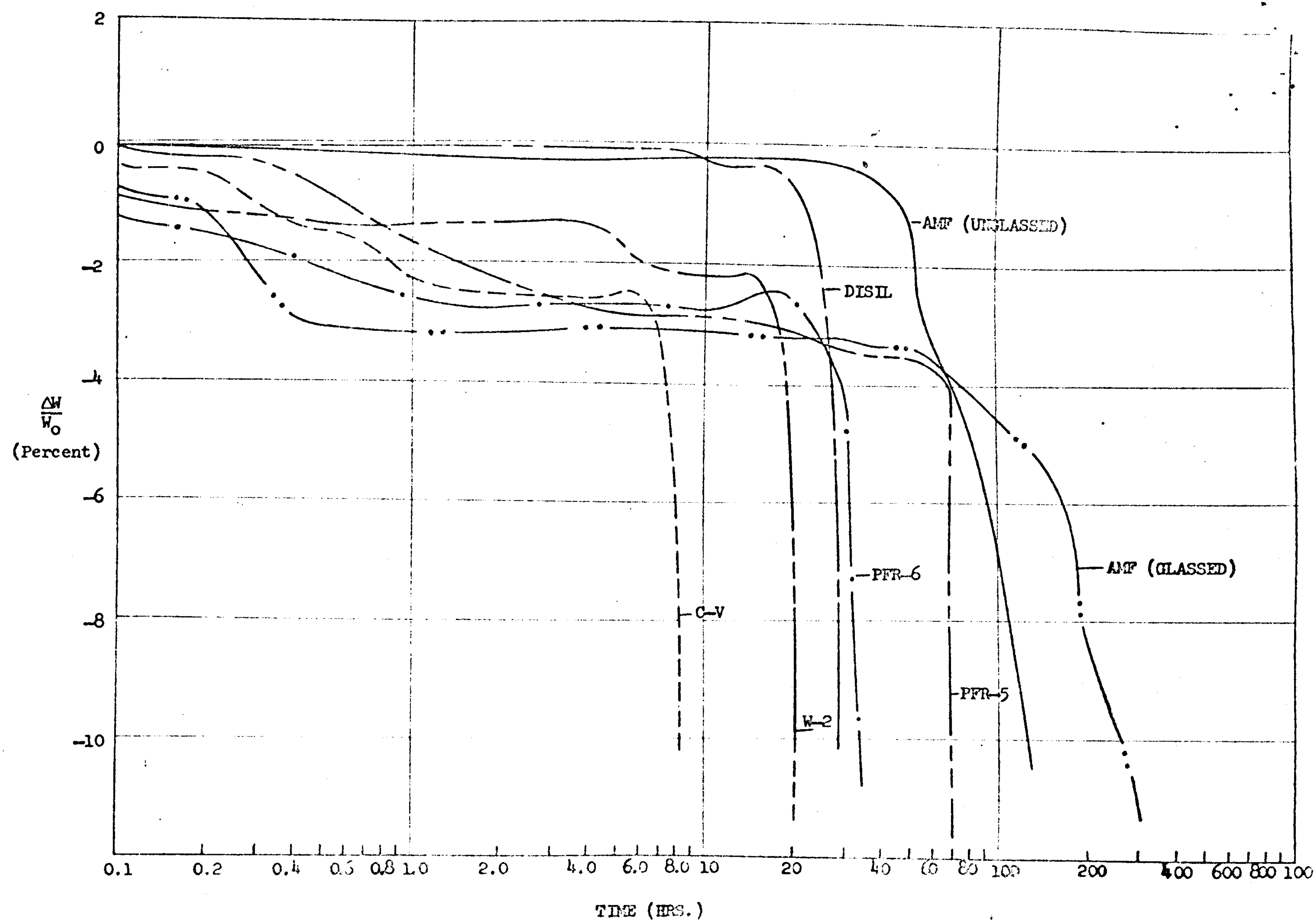


Figure 4. – TYPICAL WT. CHANGES VS. TIME FOR CONTINUOUS EXPOSURE AT 2500° F FOR COATED Mo-0.5 Ti (TUMBLED EDGES)

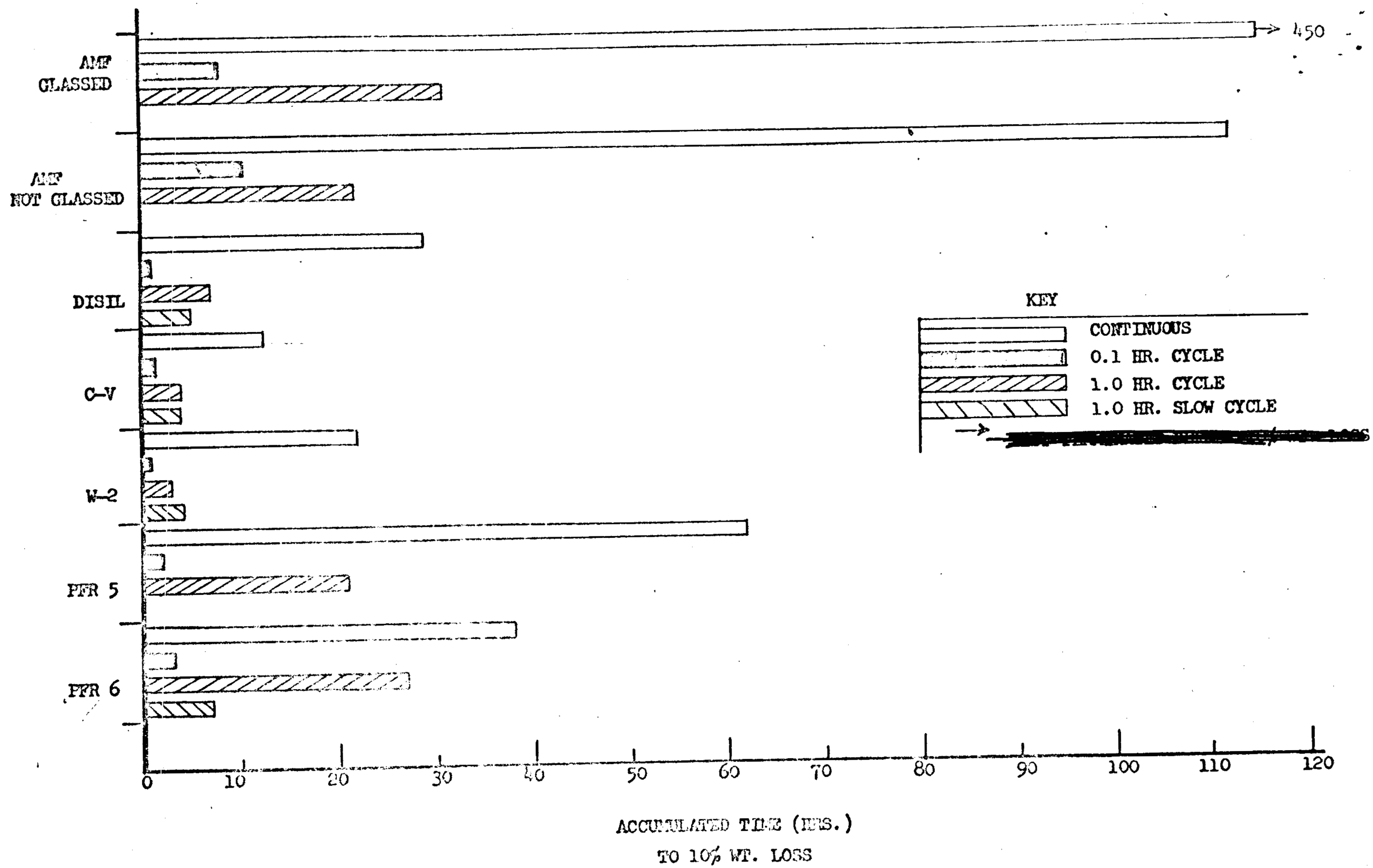


Figure 5. — COMPARATIVE RESULTS FOR CONTINUOUS AND CYCLIC EXPOSURE TESTS AT 2500° F

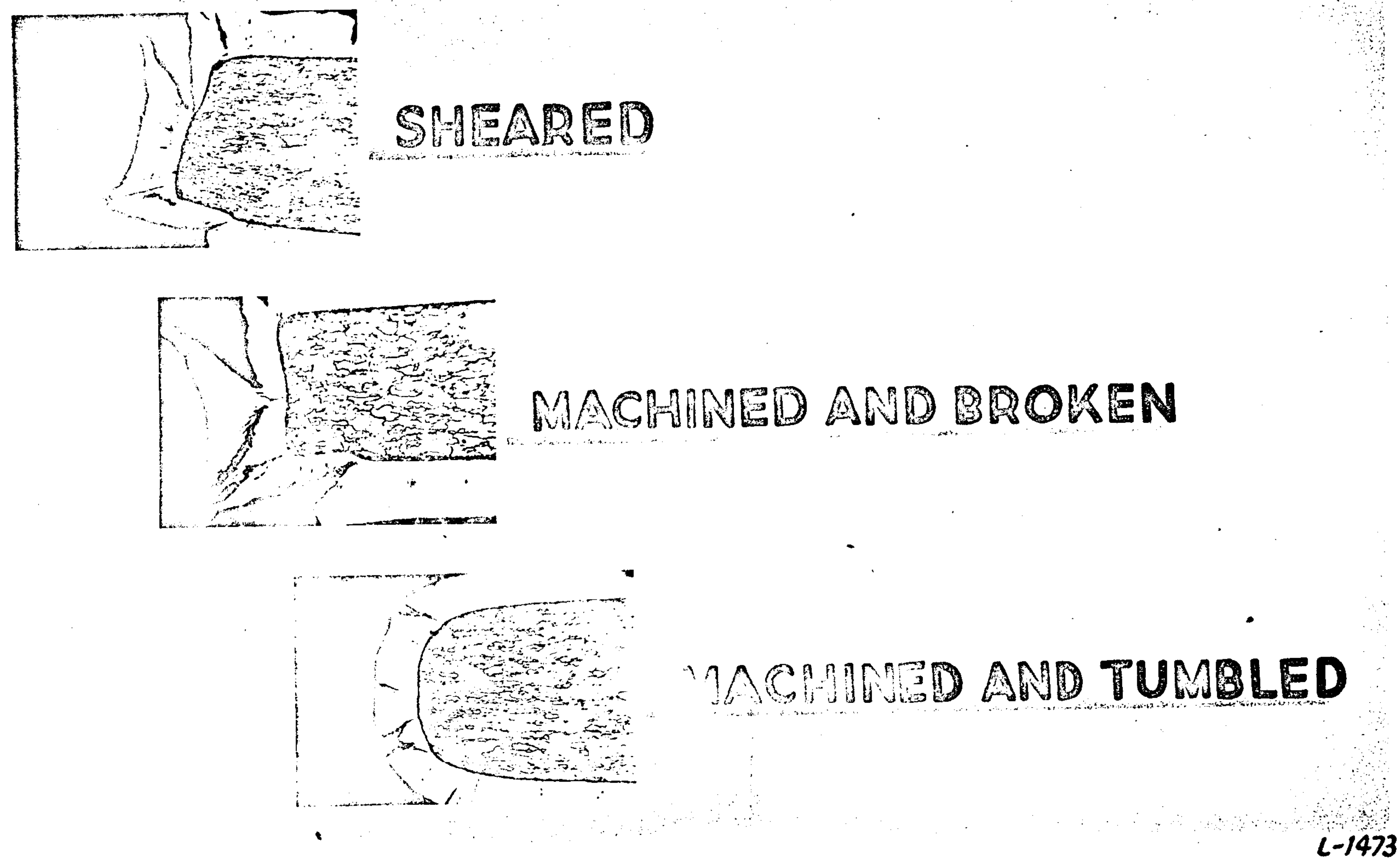


Figure 6. – COATING APPEARANCE FOR THREE EDGE CONDITIONS

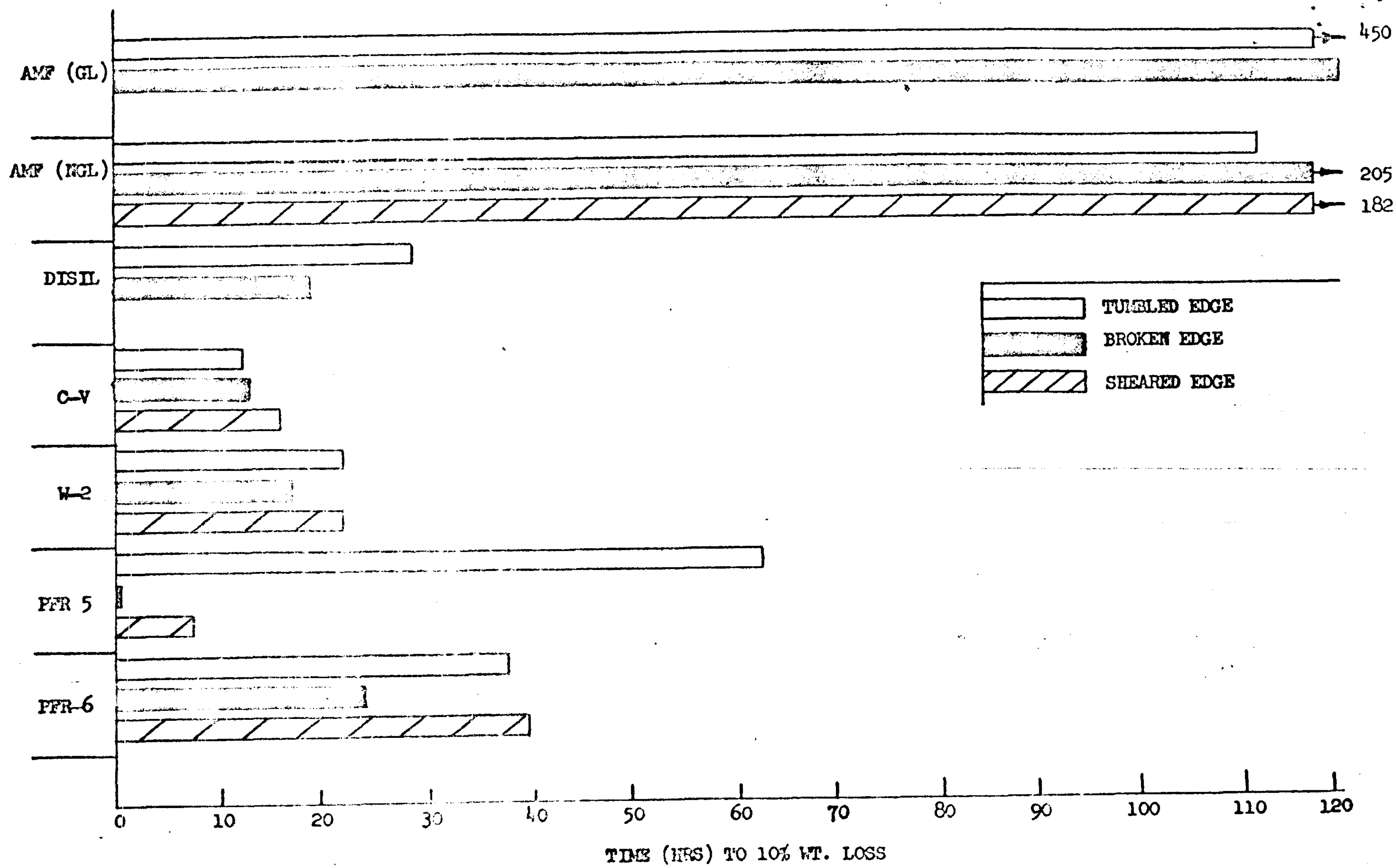
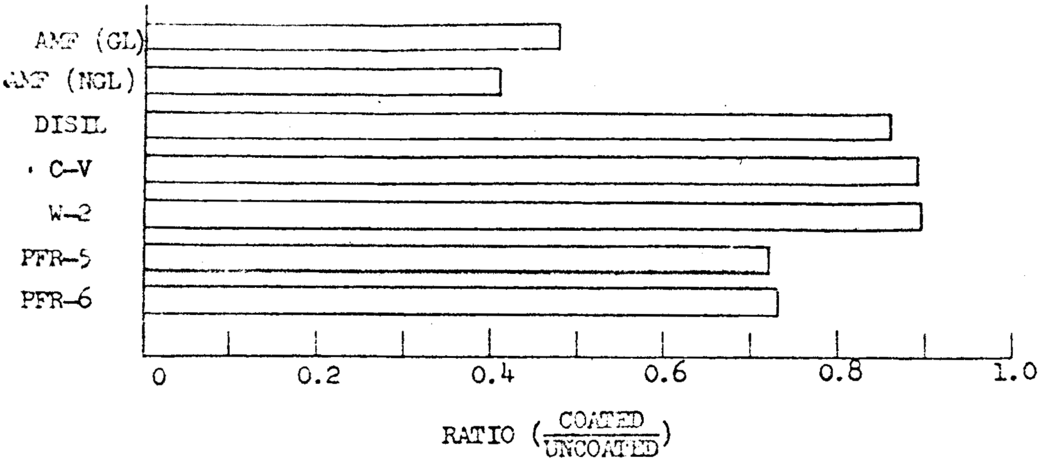
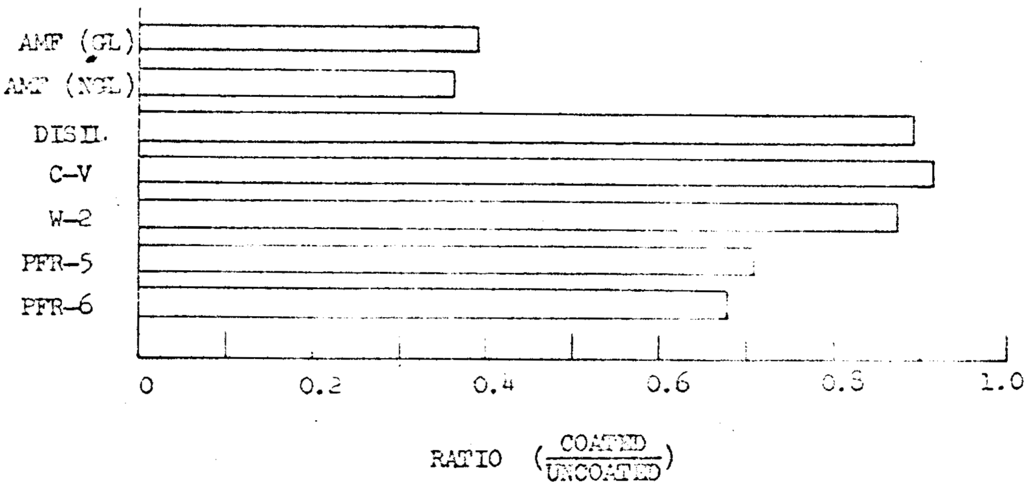


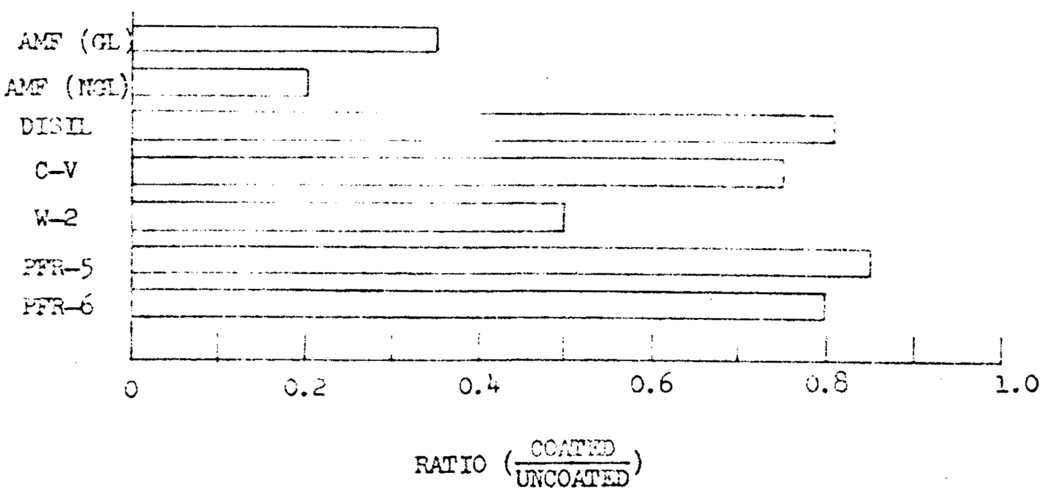
Figure 7. — CONTINUOUS EXPOSURE COATING LIFE FOR VARIOUS EDGE CONDITIONS AT 2500° F



(a) Tensile Strength



(b) Yield Strength



(c) Elongation in 2 in.

Figure 8. – RESULTS OF ROOM TEMPERATURE TENSILE TESTS

Notes: 1 – Based on Area Before Coating.

2 – Specimens Tested as Coated.