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 Glassed Titanium Specimens
 TO: M. L. Hyman
 FROM: M. L. Picklesimer

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INTRA-LABORATORY CORRESPONDENCE
Oak Ridge National Laboratory

TO M. L. Hyman

DATE May 18, 1956

COPIES TO

SUBJECT Metallographic Examination
of Glassed Titanium Specimens

Introduction

Two glassed titanium specimens were received from M. L. Hyman through W. D. Manly with the request to examine the specimens metallographically for damage to the metal. Both specimens, of A-55 titanium, were coated with a glass frit and fired at 1610°F. One specimen, called "short fire" was fired for 8 minutes, the other specimen, called "long (or slow) fire" was fired for 16 minutes.

Procedure

Metallographic specimens were cut from both samples. One specimen of each was mounted as a transverse section so that the interface between the glass coating and the metal could be examined. A second specimen of each sample was mounted with the glass-metal interface almost in the plane of polish so that the interface was "stretched out" as much as possible to permit a more detailed examination of the interface. The specimens were mechanically polished, finishing with fine diamond paste, chemically polished and etched with a solution of 50 parts glycerine, 25 parts HNO_3 , and 25 parts HF, and electrolytically anodized to form a very thin film of oxide, showing interference colors, to increase contrast in the microstructure in both bright field and polarized light examination.

Results

As the complete details of the preparation of the specimens before firing the glass frit are not available, the interpretation of the microstructure resulting from the treatment will necessarily be restricted to that which is obvious from the photomicrographs included in this report.

The microstructure of the glancing interface, long (slow) fire, specimen is shown in both bright field and polarized light in Figs. 1 and 2. The material which appears dark grey to black and which shows no particular structure in Fig. 1 (bright field) and as the bright hazy structure in Fig. 2 (polarized light) is the glass. The remainder of the structure is titanium. A thin band of relatively "clean" structure, separated from the parent metal by a dark band (Fig. 1) having an irresolvable structure at 1500X, is apparently a layer of alpha titanium containing a high percentage of oxygen, indicating that some reaction between the glass and the titanium may have occurred. The same band can also be seen in Figs. 3 and 4, the microstructures of the transverse section of the long (slow) fire specimen. The phase appearing as dark needles and as clumps in Fig. 3 is presumably either retained beta, or was beta phase at the firing temperature and, as a result of the composition or phase difference, has anodized a different color to that of the parent matrix. The "retained beta" phase appears throughout the specimen except for a band very close to the

glass-titanium interface, indicating that the firing temperature of 1610°F was within the alpha plus beta field for the material. The "retained beta", appearing as it does primarily in the grain boundaries, may or may not be detrimental to the physical properties of the glassed titanium. If this phase is retained beta, it will have acted as a "sink" for the hydrogen contained in the metal, and may, on aging at operating temperatures, precipitate the hydride in the grain boundaries, thereby embrittling the metal. These possibilities should be thoroughly checked by mechanical property testing of suitable glassed specimens. The appearance of the "retained beta" phase may be prevented by firing at a lower temperature, providing that the glass frit may be successfully fired at the lower temperature.

The microstructure shown in Fig. 5 is that of a transverse section of the long (slow) fire specimen in a region where the glass frit had been wiped off before firing. This specimen shows the oxygen rich alpha layer on the surface, the irresolvable separating band, and the "retained beta" phase discussed above. This microstructure shows that the structure of the underlying metal is not caused by reaction with the glass layer, but by the firing operation itself. However, it does not prove that there has been no reaction between the glass and the metal.

The microstructure of the glancing interface of the short fire specimen is shown in bright field in Fig. 6 and in polarized light in Fig. 7. A very careful examination of the microstructure of the entire metallurgical specimen reveals no apparent differences between the long (slow) fire and the short fire specimens. The microstructure of the transverse section of the short fire specimen is essentially the same as that of the long fire specimen shown in Figs. 3 and 4.

Conclusions

The conclusions that may be drawn from the reported examination are:

- (1) The firing temperature was within the temperature range of the alpha plus beta field of the metal, causing the formation of beta phase in the grain boundaries which either was retained as the beta phase on cooling to room temperature or decomposed to an alloy-rich alpha phase. The base material may have been embrittled by the operation.
- (2) Some reaction between the glass and the metal surface has probably occurred, but it is apparently not a serious or detrimental one.
- (3) The difference of firing times of 8 and 16 minutes has made no apparent difference in the microstructures of the two specimens, nor of the bond formed between the glass and the metal. A more conclusive statement cannot be made since only a microstructural examination has been made.

The author wishes to acknowledge the work of B. C. Leslie, Metallography Section Metallurgy Division, ORNL, in the metallographic preparation and the photomicrography of the specimens.

M. L. Picklesimer

Fig. 1 (Y-18501). Glassed Titanium. Long (slow) fire specimen. Glancing interface between glass and titanium. 500X Bright Field.

Fig. 2 (Y-18502). Glassed Titanium. Same field as Fig. 1. Polarized Light 500X.

Fig. 3 (Y-18498). Glassed Titanium. Long (slow) fire specimen. Transverse section showing interface between glass and titanium. 500X Bright Field.

Fig. 4 (Y-18500). Glassed Titanium. Same field as Fig. 3. Polarized Light 500X.

790-5

Fig. 5 (Y-18499). Glassed Titanium. Long (slow) fire specimen. Transverse section of oxidized, glass free surface. 500X Bright Field.

758-6

Fig. 6 (Y-18496). Glassed Titanium. Short fire specimen. Glancing interface between glass and titanium. 500X Bright Field.

Fig. 7 (Y-18497). Glassed Titanium. Same field as Fig. 6. Polarized Light 500X.

298 7