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October 10, 1960

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SUBJECT: EVALUATION OF COMMERCIAL CERAMIC COATINGS  
 FOR SHORT TIME PROTECTION OF COLUMBIUM  
 1% ZIRCONIUM

*Unclassified*  
 Classification

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### SUMMARY:

Fourteen commercial enamel frits from five different manufacturers were tested to determine their abilities to protect columbium 1% zirconium during the forging cycle in the temperature range of 1800 - 2300 F.

Ferro Corporation frit #3249 has been found to afford good coverage and protection at 2200 F at times up to five (5) hours exposure. Adherence was excellent during upset forging of a coated Cb/1% Zr sample heated 45 minutes at 2200 F.

Pemco Corporation frit #S-300 afforded good protection and coverage at 1700 F.

### PROCEDURE AND RESULTS:

#### I. Comparative Test

Samples of porcelain enamel high temperature frits were obtained from several manufacturers (see Table I). Each frit was mixed with water in a mortar and pestle using the following formula:

100 parts frit, 200 mesh  
 5 parts bentonite (suspension agent)  
 1 part gum tragacanth (binder)  
 1/2 part carboxy methyl cellulose,  
 medium viscosity (viscosity  
 controller)  
 80 - 90 parts water

All parts were by weight.

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CNLM 2989

Columbium 1% zirconium tabs,  $7/8" \times 1-1/2" \times .062"$ , were fabricated. The edges were rounded by grinding and the pieces abrasive blasted and cleaned with acetone.

The tabs were dip coated and dried three to four hours at 200 F. After drying, the tabs were hung on a support with type 410 stainless steel wires and placed in an electric furnace at 2200 F for one-half hour. Figure 1 shows the appearance of the tabs after removal from the furnace and cooling. Results are listed in Table I. Ferro Corporation frit #3249 was found to afford good coverage. A mixture of Pemco Corporation's frits #S-300 and #S-900 also afforded good protection but tended to pull away from corners, leaving exposed metal.

TABLE I

COATING TEST RESULTS- 2200 F FOR ONE-HALF HOUR

<u>Manufacturer</u>	<u>Frit No.</u>	<u>Remarks</u>
Ferro Corporation	XG 201	Poor
" "	3249	Excellent
" "	3251	Poor
" "	3283	Poor
" "	5210	Poor
O. Hommel Company	M-6634	Poor
" "	M-6635	Poor
Ceramic Color & Chemical Company	A-418	Poor
" " "	379-A	Poor
Pemco Corporation	R 41418	Fair
" "	S 300	Poor
" "	{ S 300 - 75% S 900 - 25% }	Good, but exposed edges
" "	S 900	Did not melt
Acheson Colloids Company	Glass in Alcohol	Poor

Pemco Corporation frit #S 300 was retested at 1700 F and afforded excellent protection. However, at 2200 F this coating ran off.

Ferro Corporation frit #3249 was selected as the best coating for use at forging temperatures and further tests were run. A piece of Cb/1% Zr bar, 2-1/4" diameter x 3-3/4" high, was coated and heated 45 minutes in air at 2200 F and upset forged in a 500 pound hammer to approximately 1-3/4" high. The piece was immediately reheated for five (5) minutes without recoating, and was upset further to 1" high. Coverage and adhesion were excellent throughout.

## II. Contamination Evaluation of Ferro #3249

Samples of Cb/1% Zr rod, 5/8" diameter x 3/4" high, were prepared. Sharp corners were broken by grinding and the samples were sand-blasted. Six (6) samples were dip-coated and dried 3 - 4 hours at 200 F.

One series of columbium samples was run uncoated at 2200 F in air for one-half hour to 5 hours. The coated series was run in the same manner. Figure 2 shows the appearance of an uncoated and a coated piece after 5 hours in air at 2200 F.

After heat treatment the samples were cut in half, mounted, and metallographically polished. Figure 3 shows 400 gram Knoop hardness profiles for uncoated and coated samples at times up to 5 hours. The uncoated samples attained maximum hardness quickly. This hardness, equivalent to  $R_c$  44-46, appears to be the maximum attainable. The metal then turns to oxide; the surface receding at a linear rate of approximately .011 inches/hour. The coated samples never attained a surface hardness greater than 250 KHN ( $R_B$  100), although the depth of contamination increased with time.

## III. Heating Time for Coated Billet

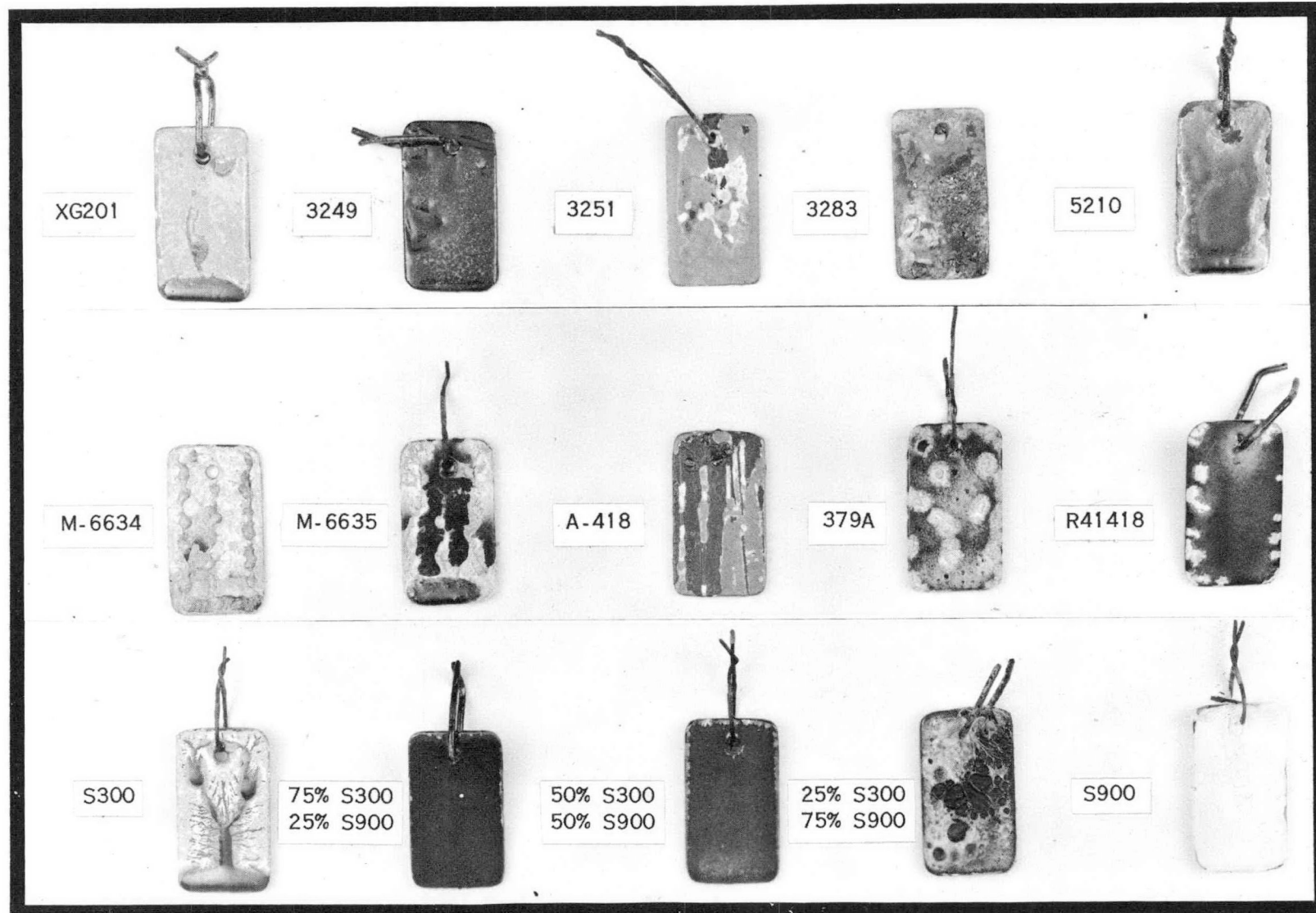
A Cb/1% Zr billet, heat code PHGP, 11" diameter x 10" long was drilled radially at mid-height to accommodate thermocouples at depths from 2" to center. The billet was coated with Ferro #3249, dried, and placed in a 2200 F electric furnace on a mild steel tray. Figure 4 shows that surface and center temperature were equal after 45 minutes. This data shows that current heating cycles for full scale forgings are quite adequate. Further usage of this data will be for predicting heating times for various size ceramic coated billets.

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DWM:bep

# CB-1 Z<sup>R</sup> TABS COATED WITH CERAMIC FRITS

SEE TABLE 1 FOR MANUFACTURERS NAMES



FERRO CORPORATION FRIT NO. 3249

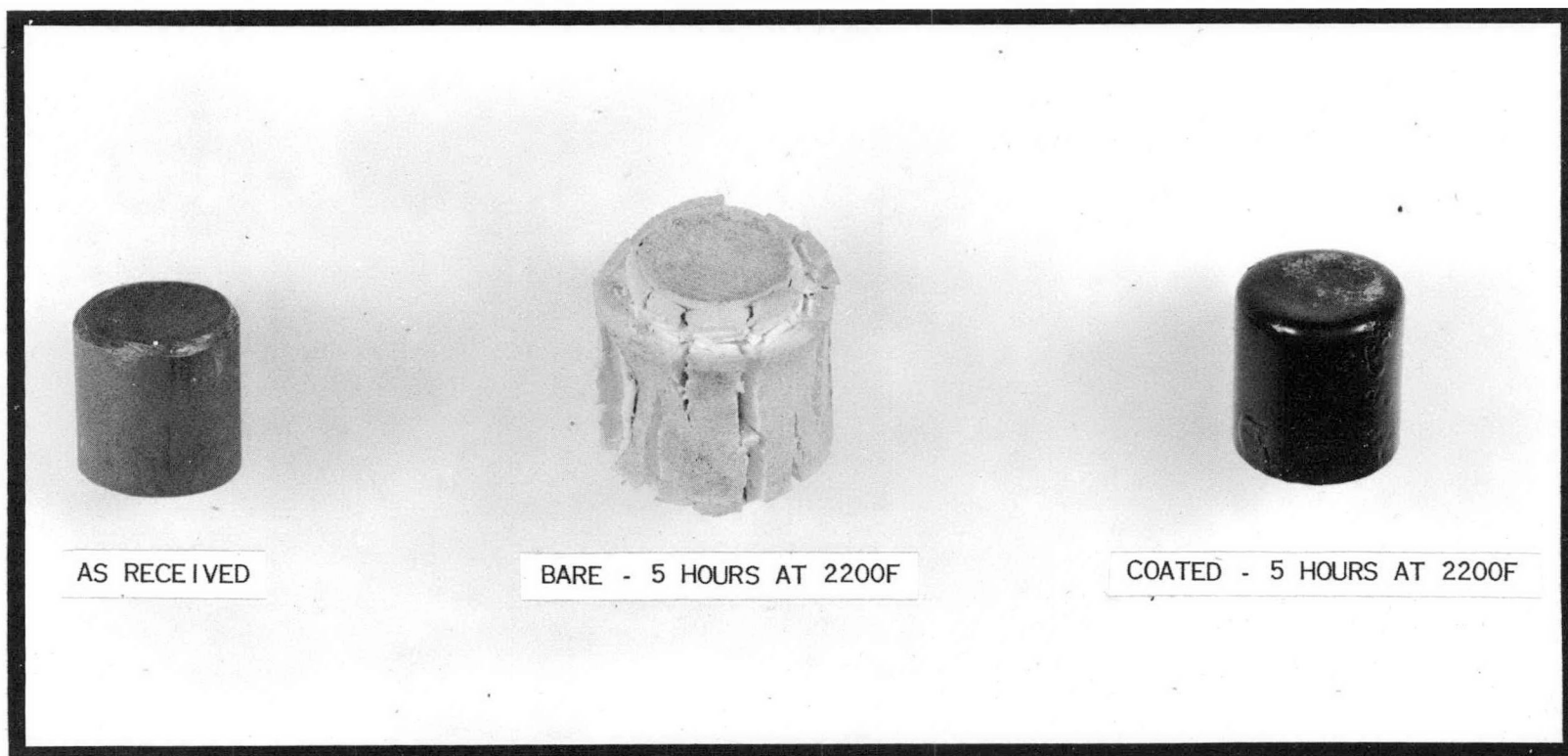


FIG 2

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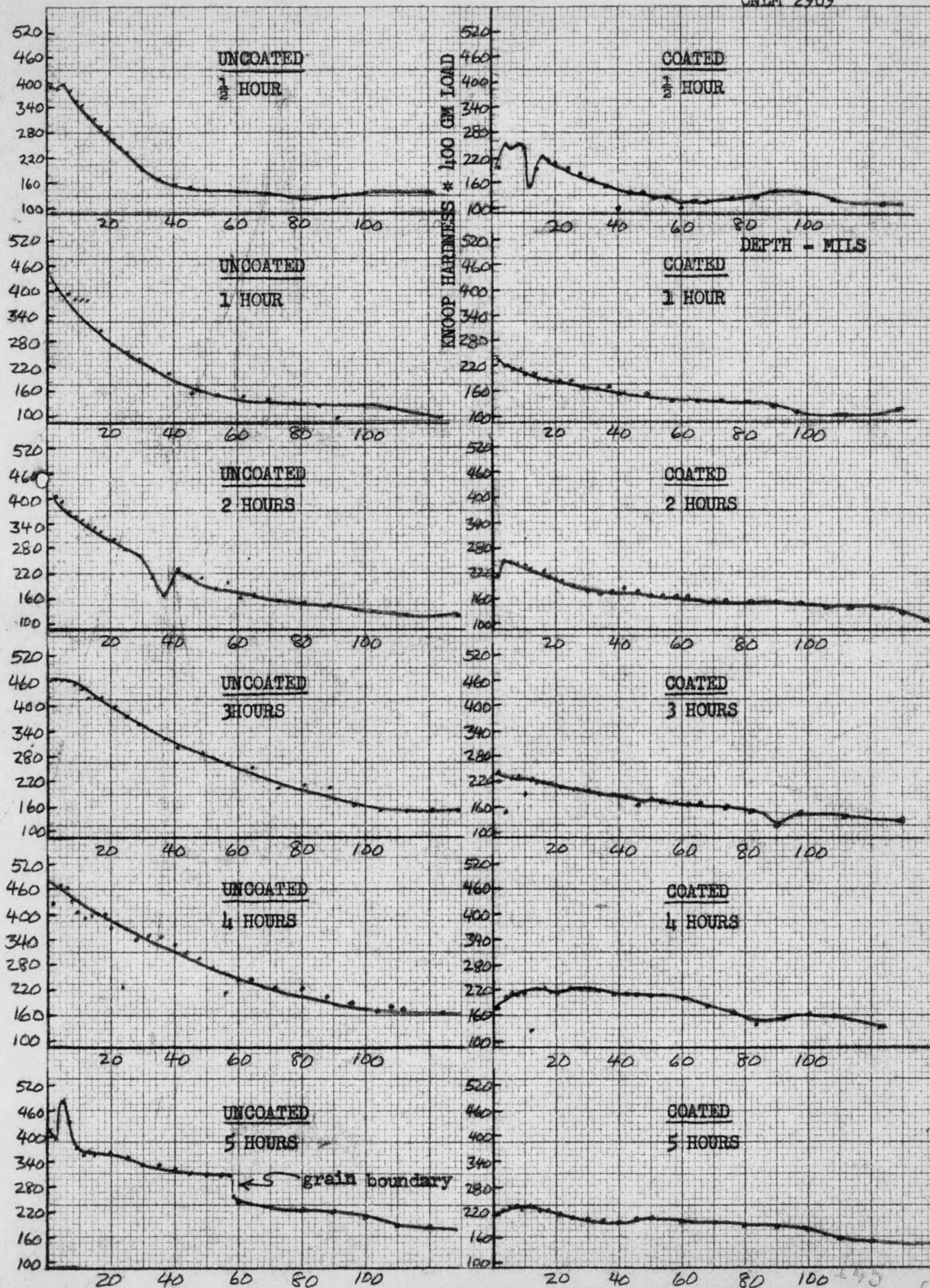


FIGURE 3. HARDNESS PROFILES OF UNCOATED AND COATED SAMPLES, 2200 F IN AIR.

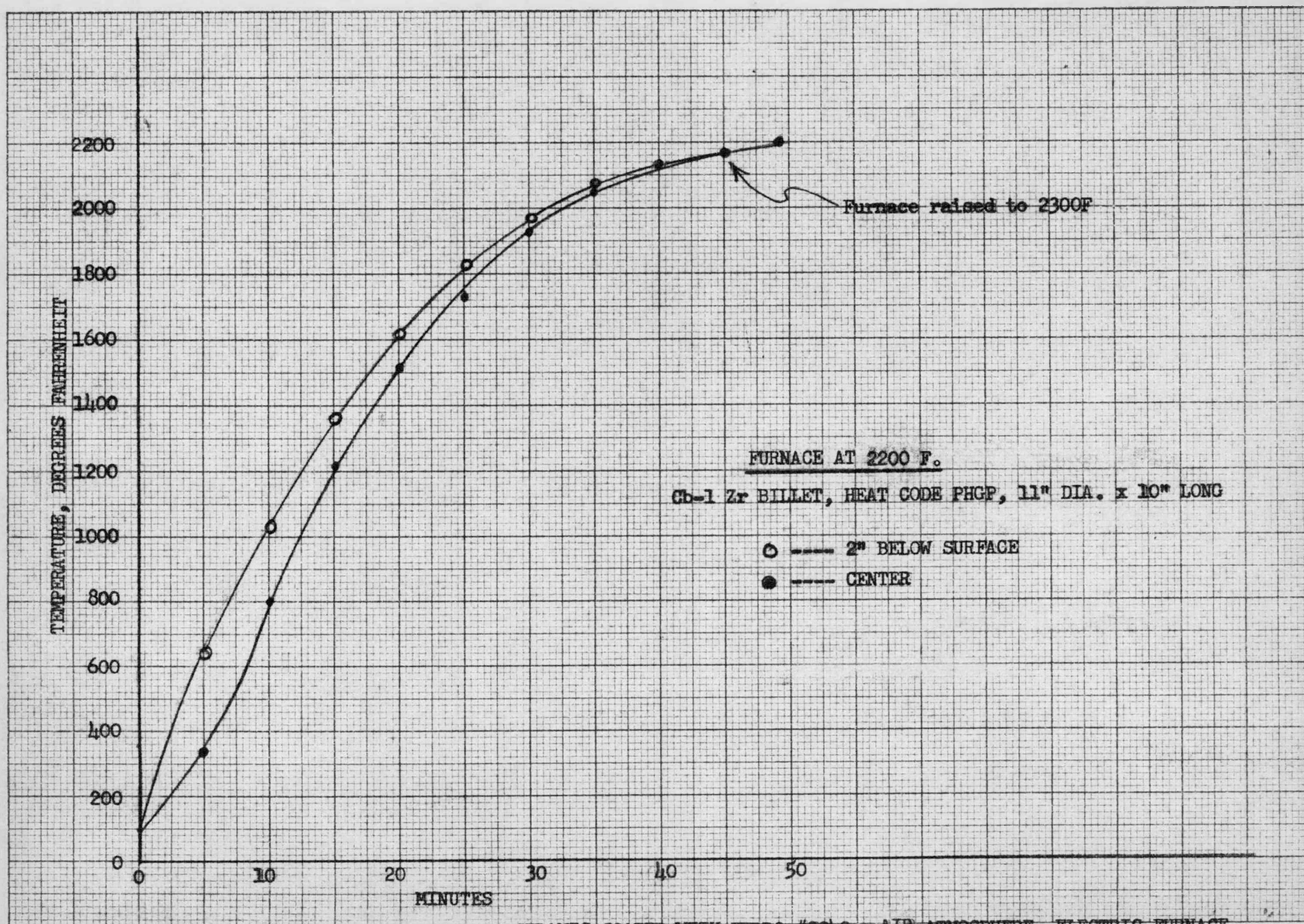


FIGURE 4. HEATING TIME FOR BILLET CERAMIC COATED WITH FERRO #3249. AIR ATMOSPHERE, ELECTRIC FURNACE.