THERMAL CONDUCTIVITY AND THERMAL SHOCK QUALITIES OF ZIRCONIA COATINGS ON THIN GAGE HASTELLOY-X METAL

By John D. Buckley

NASA Langley Research Center
Langley Station, Hampton, Va.

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SUMMARY

Two zirconia coatings and a zirconia-nickel aluminide cermet coating were flame sprayed and plasma sprayed on thin-gage Hastelloy-X metal. Thermal insulation measurements were made on a series of specimens to determine the relationship between increased coating thickness and thermal insulation capacity of these three coatings. Thermal shock measurements were conducted to observe the effect of coating application and/or thickness on adherence of coatings to the Hastelloy-X metal surfaces. The results showed that composition and mode of application influence thermal shock resistance of sprayed coatings. Insulation capacity of the sprayed coatings proved to be linearly related to coating thickness.

INTRODUCTION

A matter of concern in the design of aerospace vehicles is the insulation of critical metal structures from the extreme heat fluxes and temperatures produced by flight at hypersonic velocities (ref. 1, 2, and 3). Ceramic coatings, particularly those with low heat transfer coefficients and high melting points, are very good thermal barriers. Stabilized zirconia, one of the highest melting refractory oxides ($\simeq 4600^\circ F$) (ref. 4), has a very low heat transfer coefficient

(15 Btu/hr. ft.^{2} ^{o}F/in.) (ref. 5). It has, however, only fair thermal shock resistance when compared to that of other ceramic refractories (ref. 4 and 6).

Flame spraying and plasma spraying are two methods used to apply protective coatings on metals that are subject to extreme thermal environments (ref. 7 and 8). Stabilized zirconia coatings have been used as thermal protection in rocket nozzles and have been considered for use as a heat barrier on the leading edges of aerospace vehicles (ref. 9 and 10). The objectives of this study were: (1) to observe the effect of thermal shock on coating thickness for zirconia and a zirconia cermet flame sprayed and/or plasma sprayed on Hastelloy-X; and (2) to determine the relationship between increased coating thickness and thermal insulation capacity of the zirconia coatings on Hastelloy-X.

MATERIALS AND SPECIMENS PREPARATION

The materials evaluated in this study are presented in table 1, and the specimen configuration is shown above the table. The spray compositions evaluated included stabilized zirconia and a zirconia-nickel aluminide cermet. Five chromel-alumel thermocouples were welded on each of the 4 square inches by 0.02-inch thick Hastelloy-X specimens prior to coating. Coatings, applied by the NASA Langley Research Center fabrication shop, were sprayed on the side opposite the thermocouple locations. Before spraying, specimens were cleaned with methyl ethyl ketone and sandblasted with 120-grit aluminum oxide. A 0.003-inch base coat of nickel aluminide (80 weight percent Ni, 20 weight percent Al) was plasma sprayed on each specimen before the insulating coat was applied. Seven specimens were made for each of the three protective coatings evaluated. Total coating thickness varied between 0.006 and 0.024 inch, applied in increments of 0.003 $\pm$0.001 inch (table II).

Stabilized zirconia rod was used for the flame spraying operation, while powdered stabilized zirconia and powdered zirconia-nickel aluminide cermet were used for plasma spraying.

EQUIPMENT AND TEST PROCEDURE

Figure 1 shows the equipment used in obtaining the thermal shock and thermal conductivity data. Each specimen was fastened into an inset in a block of masonite in order to present an even or smooth surface to the plasma torch and airjet. This fastening method minimized the flame impingement from the plasma torch and cold air from the airjet around the edge and onto the back surface of each specimen. The five thermocouples welded to the back of each specimen were attached to a multichannel oscillograph to record changes in the cold-face temperatures of each specimen when subjected to cyclic heating. The temperature of the uncoated hot face of the Hastelloy-X and the coated surface of each sprayed specimen was measured with a Barnes Engineering Company, model no. R4F1, total radiation pyrometer using a filter which transmitted wavelengths between 4 μ and 15 μ. An emittance value of 0.84 was used for the Hastelloy-X (ref. 11), 0.8 for the ZrO_2 coatings (ref. 5), and 0.84 for the cermet (ref. 11). Heating rates were measured by a metal calorimeter made of Hastelloy-X having the same dimensions as the Hastelloy-X specimens that were coated.

Thermal Shock Tests

Thermal shock resistance of the three candidate coatings was determined by submitting each specimen to 50 quick heating and cooling cycles. Every specimen was subjected to the direct heat of a plasma torch which produced a heat flux of 55 Btu/ft.^{2} sec. followed by immediate quick cooling with ambient air at 80 psi. One cycle took 80 seconds and consisted of the following steps: (1) Programmed the movement of the plasma torch on a track from a position 6 inches clear of the specimen, onto the specimen at a speed of 2.5 seconds per inch; (2) held the torch on the specimen for 20 seconds; (3) moved the plasma torch from the center of the specimen to its initial position, 6 inches clear of the specimen at a speed of 2.5 inches per second; and (4) automatically cooled the specimen with a blast of compressed air for 30 seconds.

Thermal Insulation Tests

Thermal insulation data was obtained simultaneously while the thermal shock tests were being conducted. Temperature measurements were recorded during each cycle from the thermocouples located on the back surface and from the total radiation pyrometer aimed at the center of the coated front surface of each specimen. The insulation quality of the coatings was determined by comparing cold-face temperatures of the various coated specimens with an uncoated Hastelloy-X standard, and also by measuring temperature differences between the radiation pyrometer readings on the coated surface and the thermocouple located on the cold-face of the specimen immediately behind the radiation pyrometer target area.

RESULTS AND DISCUSSION

Thermal Shock Tests

Thermal shock tests were made on the Hastelloy-X specimens which were coated with zirconia and with zirconia-nickel aluminide cermet to determine if coating thickness and/or method of application affected degree of spallation.

Figure 2 presents a plot of a typical thermal shock cycle. This curve was generated from total radiation pyrometer readings taken on the hot-coated surface of several typical specimens. It shows that these coatings experienced temperature changes of approximately 1700°F in 20 seconds when cycled between 200°F and 1900°F.

Photographs (figure 3) of specimens subjected to thermal shock show that after 50 cycles, the flame-sprayed stabilized zirconia and the plasma-sprayed zirconia-nickel aluminide cermet were superior to the plasma-sprayed zirconia specimens. It was also noted that spallation of plasma-sprayed zirconia coating was independent of coating thickness.

Fracture of the zirconia plasma-sprayed coating is believed to have resulted because of the inherent good-to-fair thermal shock quality of zirconia (ref. 4) compounded by the greater density characteristic of plasma-spray coatings as compared to flame-sprayed zirconia coatings (ref. 9). The good thermal shock quality of the cermet spray coating is believed to result from the better thermal conductivity of the nickel and aluminum components in the cermet spray composition (ref. 12).

Thermal Insulation Tests

The thermal insulation capacity of the three spray coating compositions was evaluated at elevated temperatures by (1) measuring variation of back- or cold-face surface temperature with increasing coating thickness, (2) measuring differences in temperature through the thickness of each of the coated specimens, and (3) determining the thermal conductivity of each specimen as a function of coating thickness for each spray coating composition.

The results of the cold-face surface temperature measurements, taken on the uncoated and coated specimens, are presented in figure 4. Data from the zirconia plasma-sprayed specimens was too erratic to be plotted. It is believed that the cracked and/or fractured coatings on these specimens caused the scatter observed in these temperature measurements.

The curves in figure 4 show that the back- or cold-face temperature of the coated specimens does decrease with increasing coating thickness, that the temperature drop is linear for both coatings, and that the difference in cold-face temperature is greater for the zirconia flame-spray coating than for the cermet-coated specimen having approximately the same coating thickness.

Figure 5 shows curves of temperature difference as a function of total coating thickness for the flame-sprayed zirconia and the plasma-sprayed cermet. The temperature difference between the hot and cold faces of the flame-sprayed zirconia and plasma-sprayed cermet increases linearly with coating thickness. The temperature difference between the hot and cold faces of the flame-sprayed zirconia coating compared to the plasma-sprayed cermet showed the temperature drop through the flame-sprayed zirconia specimen to be 35°F to 45°F greater than that through the cermet-coated specimen having approximately the same coating thickness.

Thermal conductivity values calculated from experimental data obtained during the cyclic heat tests showed that the coatings evaluated produced a very effective thermal insulating barrier (table III). Variation in coating thickness showed little or no effect on the coefficient of thermal conductivity for either of the spray coating compositions. The zirconia flame-spray coating had the lower coefficient of thermal conductivity and, thus, was the better insulator.

Figure 6 exhibits graphically a comparison between the temperature drop through uncoated metal and metal coated with 0.024 inch of zirconia flame-spray and cermet plasma-spray coatings. This data, combined with the data from table III, shows that the flame-sprayed zirconia coating is a good thermal barrier for thin-gage Hastelloy-X metal.

CONCLUDING REMARKS

The results of the evaluation of the thermal shock and thermal insulation qualities of the flame-sprayed zirconia coating, the plasma-sprayed zirconia coating, and the plasma-sprayed zirconia-nickel aluminide cermet coating are as follows:

1. Flame-sprayed zirconia coating has better thermal shock resistance than plasma-sprayed zirconia coating of similar composition and coating thickness.

2. The spallation of plasma-sprayed zirconia coatings is independent of coating thickness up to the 0.024-inch thick coating tested.

3. Flame-sprayed zirconia coating is a better thermal insulator than the plasma-sprayed cermet coating.

4. Insulation capacity of flame-sprayed zirconia coating on Hastelloy-X metal is a linear function of thickness.

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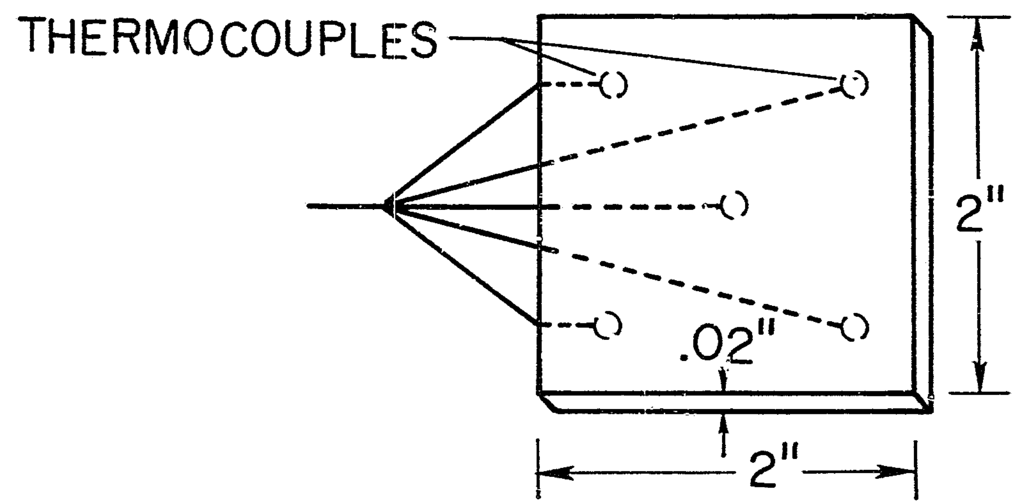
TABLE I

SPECIMEN CONFIGURATION



MATERIALS

GRADE	ANALYSIS, WEIGHT PERCENT OF									
	ZrO_2	Al_2O_3	SiO_2	Fe_2O_3	TiO_2	NaO_2	CaO	HfO_2	Ni	AL
STABILIZED ZrO_2(FLAME)	92.57	.63	.33	.33	.39	.02	3.73	2.0		
STABILIZED ZrO_2(PLASMA)	93.0		.35	.5			5.0	1.15		
ZrO_2 CERMET (PLASMA)	32.6			.17			1.7	.41	52.0	13.0
NICKLE ALUMINIDE									80.0	20.0

TABLE II

SPRAY COATING THICKNESS

SPEC. NO.	BASE COAT, in.	INSULATING COAT, in.	TOTAL COAT, in.
1	.003	.003	.006
2		.006	.009
3		.009	.012
4		.012	.015
5		.015	.018
6		.018	.021
7		.021	.024

TABLE III
THERMAL CONDUCTIVITY

	$K = Btu/ft^2\ hr\ {}^\circ F/in.$
HASTELLOY X UNCOATED	203.5* (ref. 11)
HASTELLOY X + CERMET COATING	30.3
HASTELLOY X + FLAME SPRAY ZrO_2 COATING	21.8

*EXTRAPOLATED

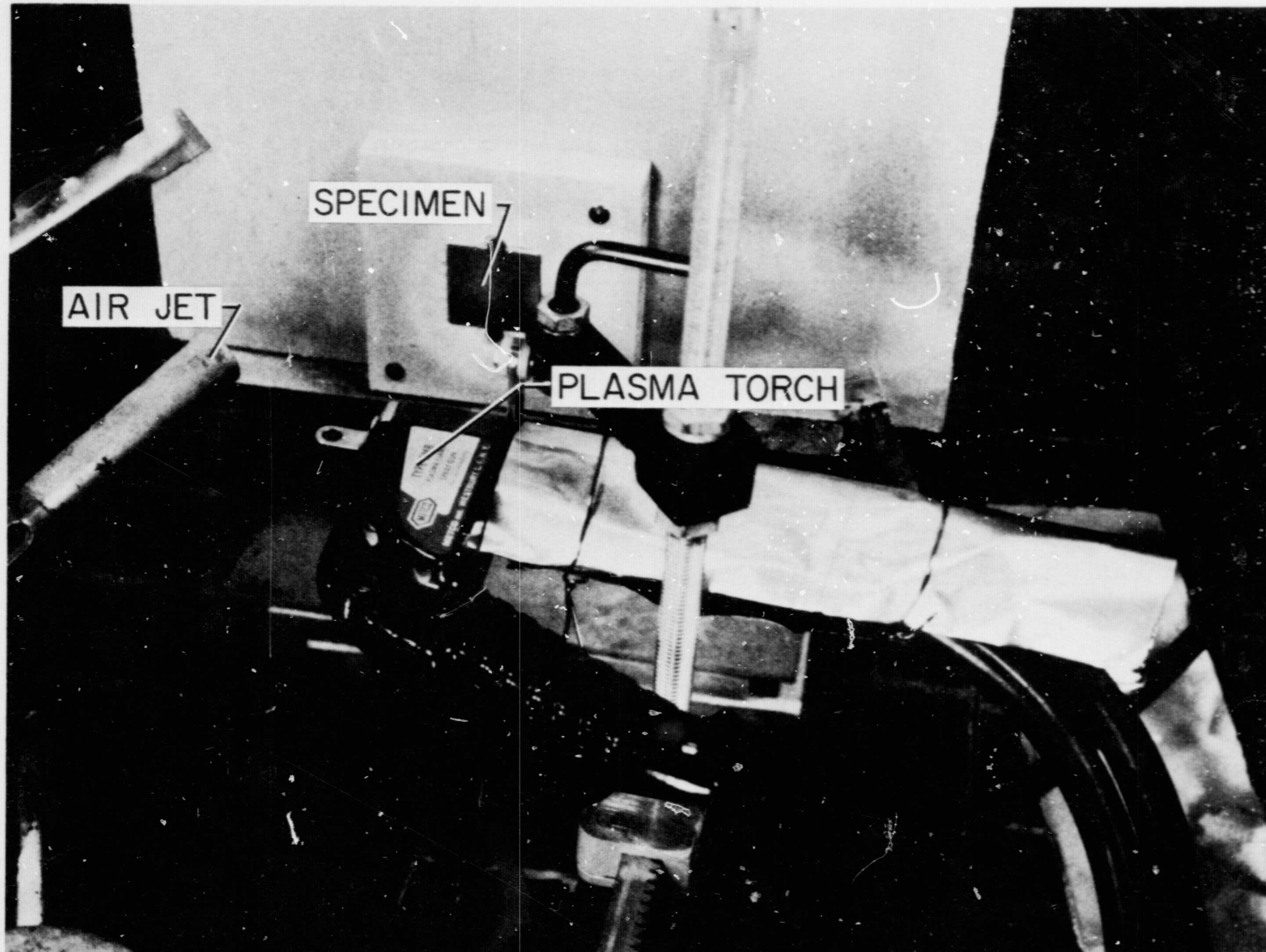


Figure 1.- Thermal shock and thermal insulation test equipment.

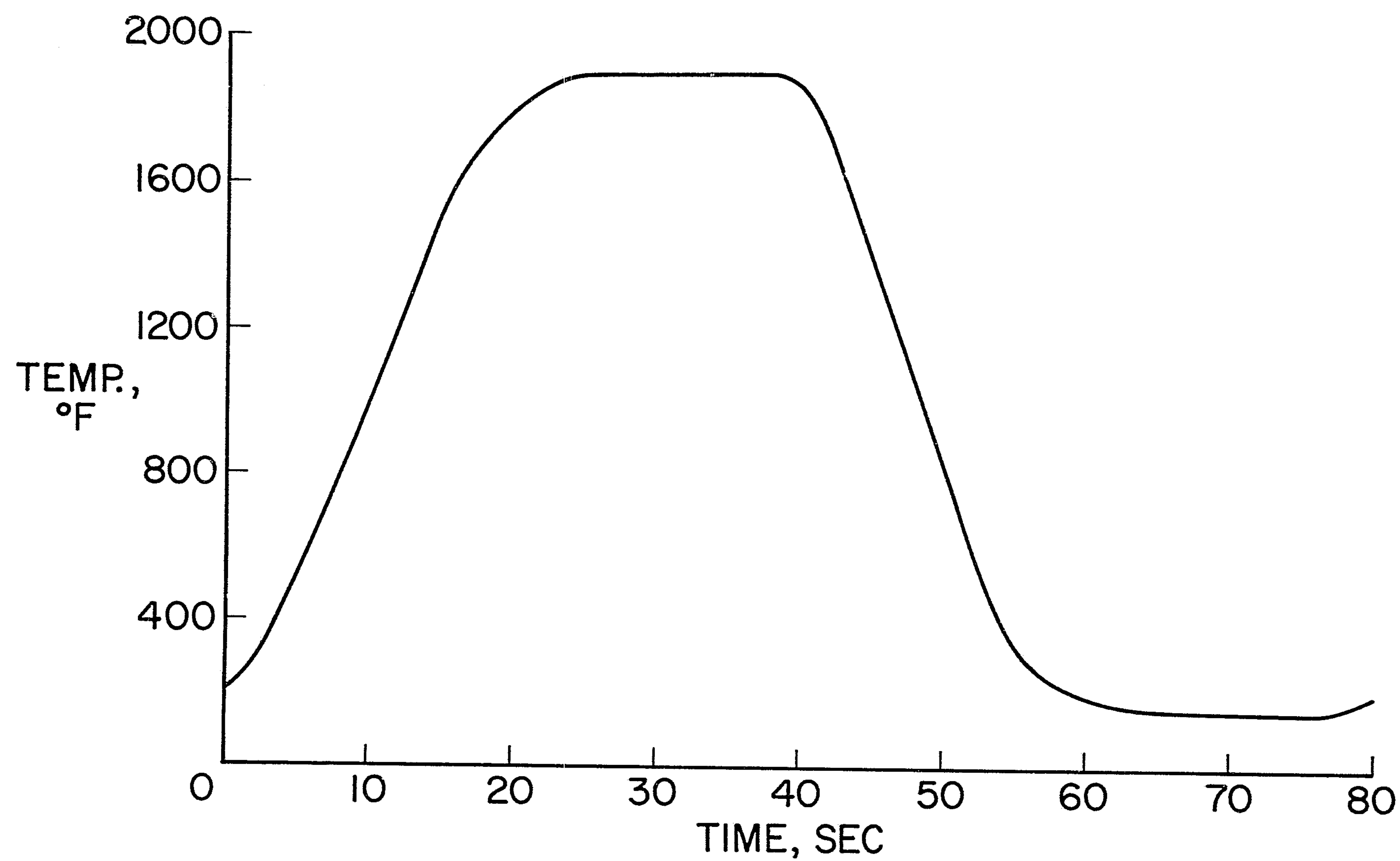


Figure 2.- Typical thermal cycle for the thermal shock and insulation tests.

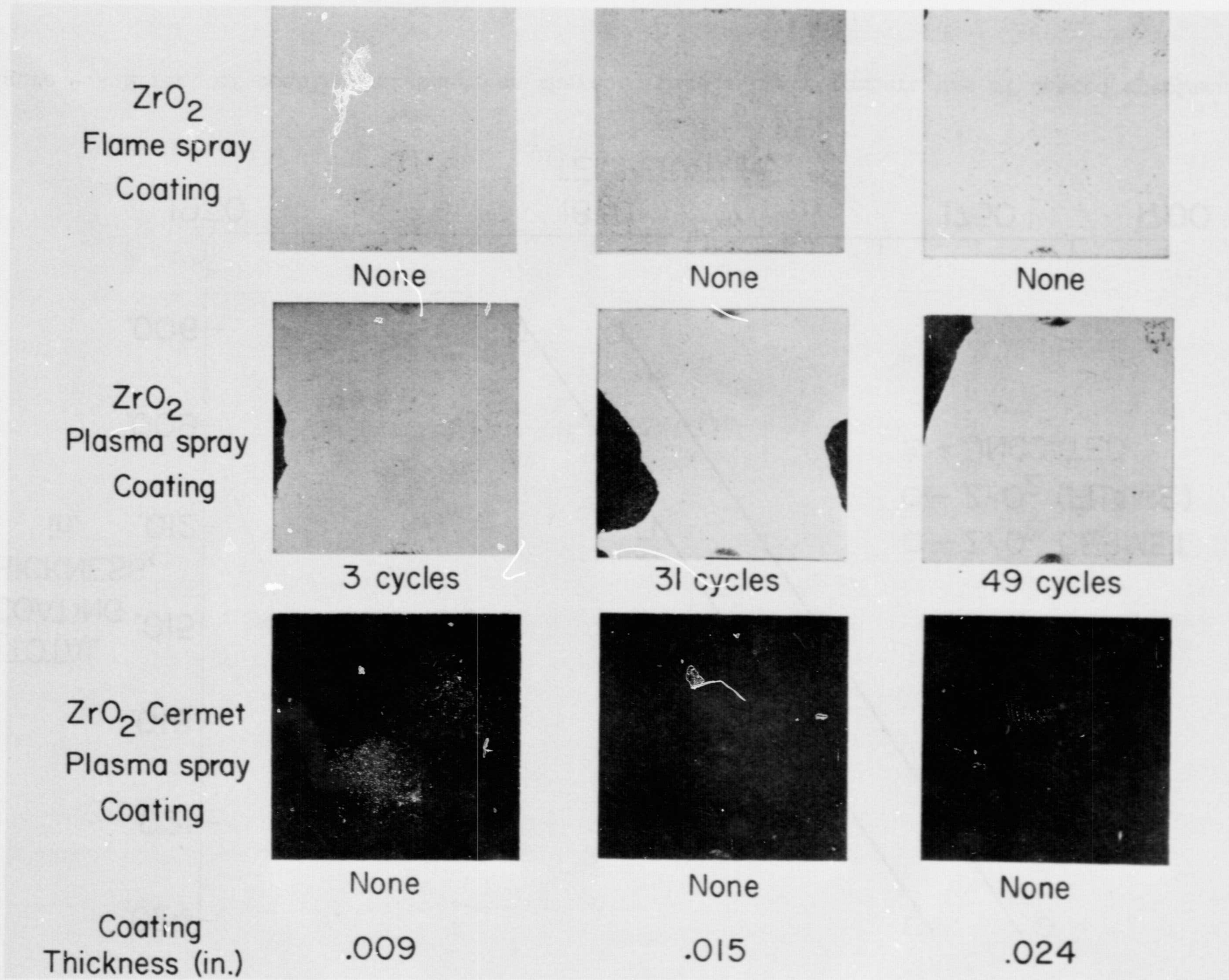


Figure 3.- Photographs of Hastelloy-X with ZrO_2 plasma and flame spray specimens and cermet plasma spray specimens subjected to 50 thermal shock cycles. Δt on heating and cooling was $\simeq 1700^\circ$ F in 20 seconds. (Cycles to initial spallation are indicated.)

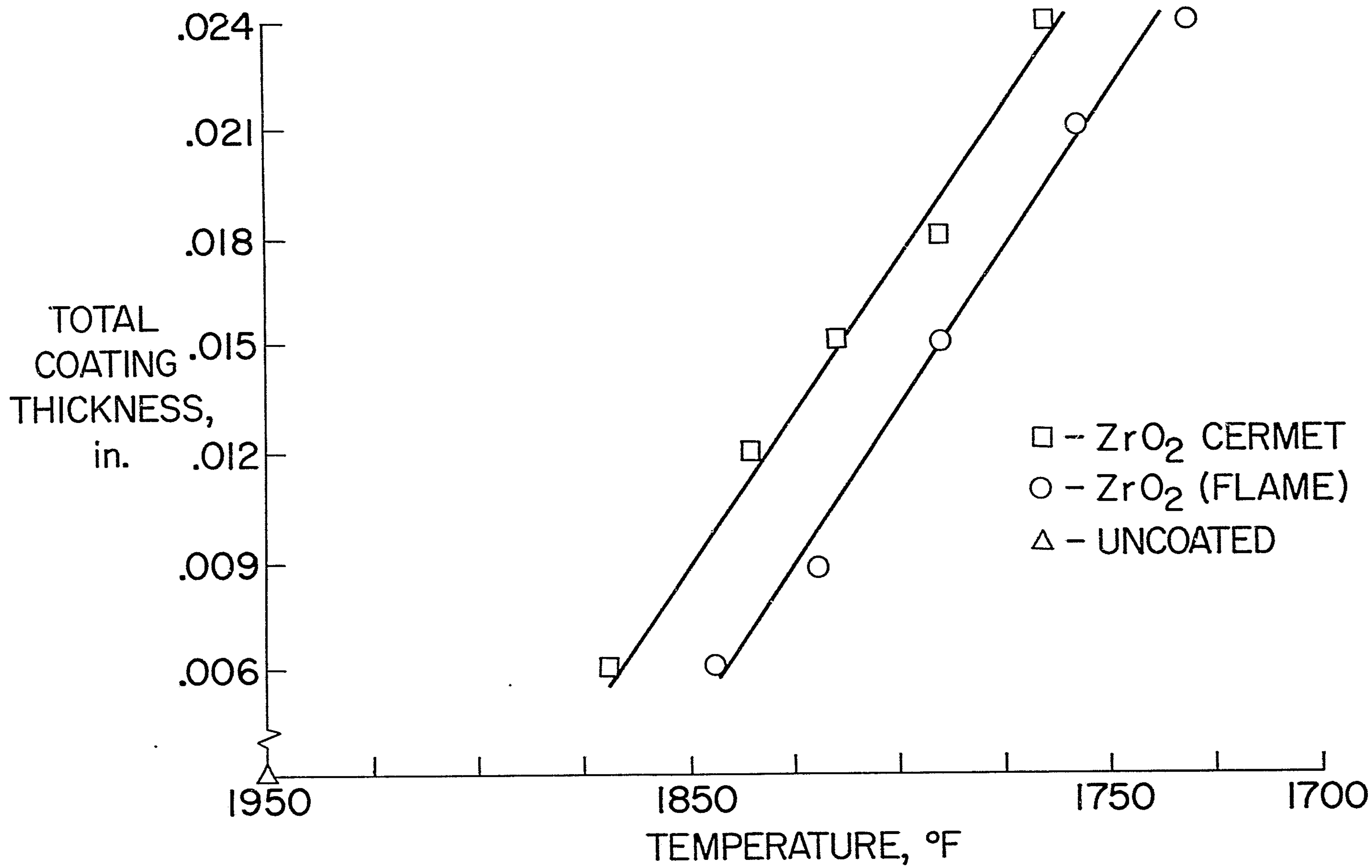


Figure 4.- Effect of coating thickness on the cold face surface temperature of coated specimens.

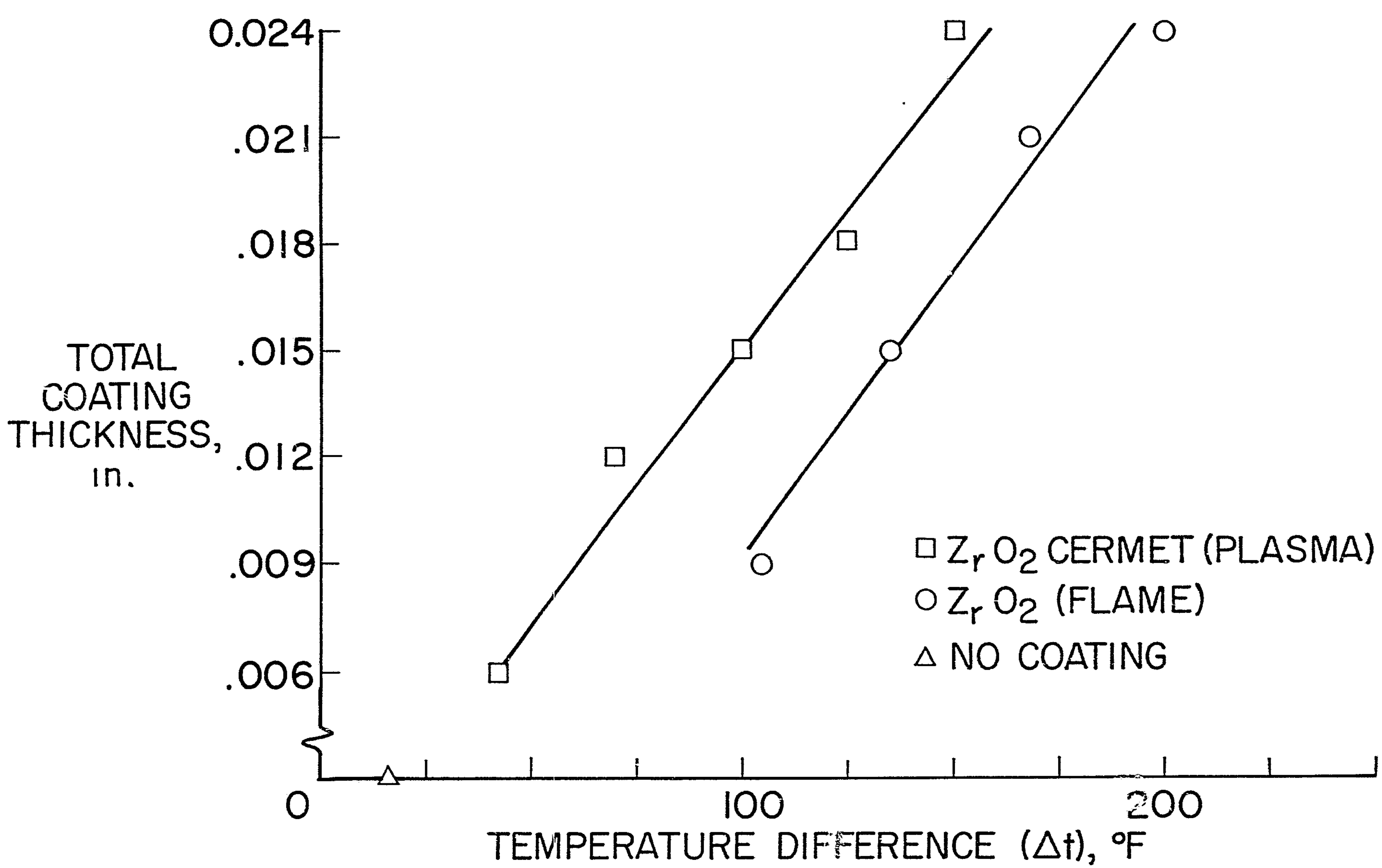


Figure 5.- Temperature drop through coated specimens as a function of coating thickness.

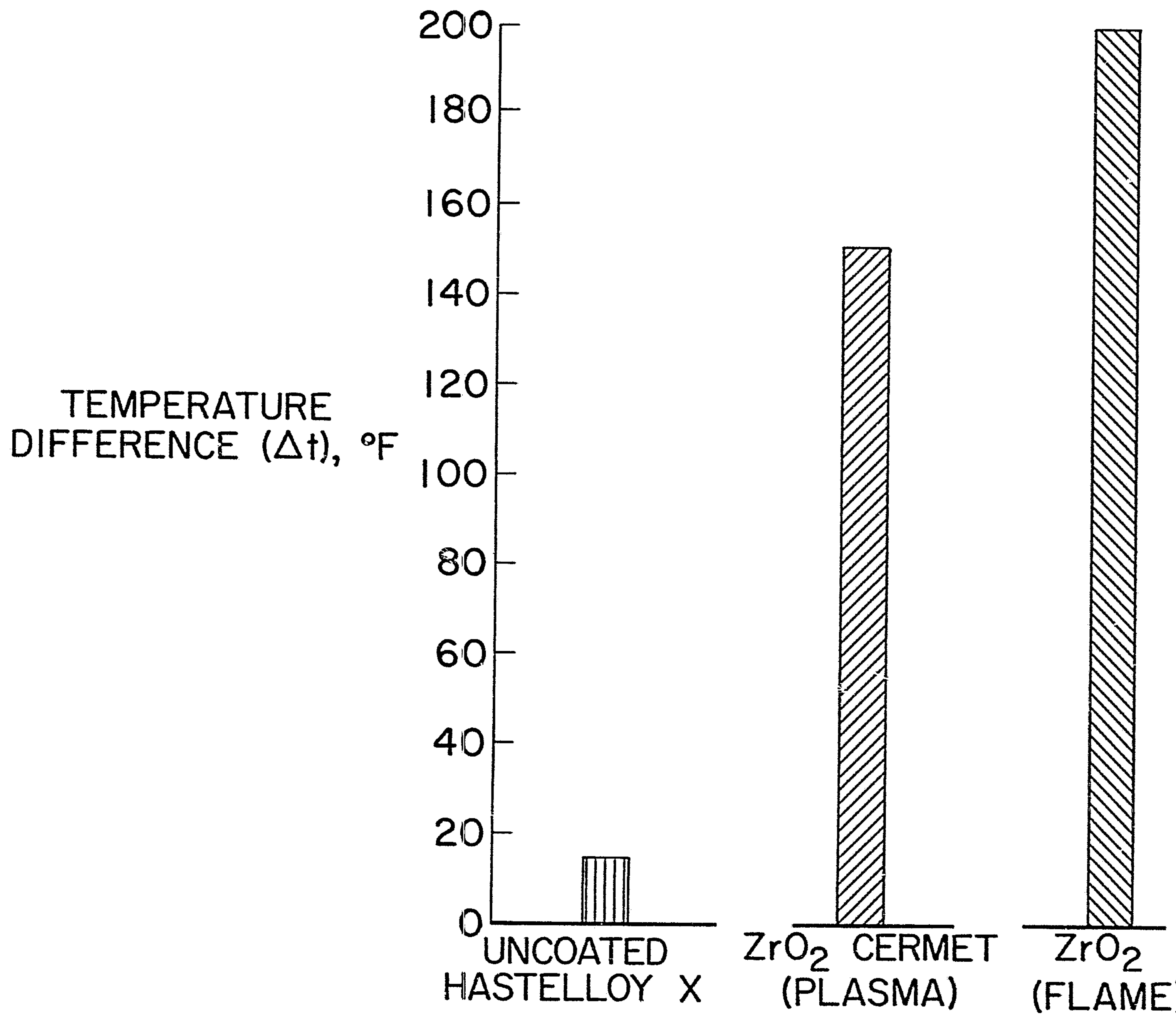


Figure 6.- Comparison of thermal drop of uncoated metal and coated metal 0.024-inch thick.

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