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EFFECTS OF PREOXIDATION TREATMENTS ON SPECTRAL NORMAL AND TOTAL NORMAL EMITTANCE OF INCONEL, INCONEL-X, AND TYPE 347 STAINLESS STEEL

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SUMMARY

The spectral-normal-emittance values of several oxidized surfaces prepared by varying the preoxidation treatments or oxidation time for inconel, Inconel-X, and type 347 stainless steel were determined at temperatures of 900°, 1,200°, 1,500°, and 1,800° F over a wavelength range of 1 to 15 microns. Polishing, grit blasting, etching, or combinations of these preparations were used as preoxidation treatments. These values were compared for 900° and 1,800° F to determine the effects of these treatments on the spectral-normal-emittance values.

Significant effects of preoxidation treatments and oxidation times on the spectral normal emittances of oxidized inconel, Inconel-X, and type 347 stainless steel are presented. In general, if a grit-blasted surface is etched before being oxidized, the final oxidized surface will have a lower emittance but will be more adherent and uniform. Of the two types of grit used in this study, the coarser grit provided the higher emittance. Polishing provided the lowest emittance of all specimens tested. In the one set of tests in which oxidation time was varied (on the inconel specimens), increasing oxidation time increased the emittance; however, increasing the time beyond 2 hours produced no further effect.

INTRODUCTION

A considerable quantity of emittance data is available for use by designers, but for many materials there are large differences between the data obtained by different investigators testing the same material. The variations arise from problems inherent in the methods of measurement, unknowns in the composition of materials, contamination of surfaces, and varying techniques for preparation of the surfaces. The most common source of discrepancies appears to be the last of these, namely, the lack of accurately defined, reproducible surfaces whose preparation and method of testing is clearly and accurately described. Information on the reproducibility of a surface, specifications describing in detail the exact surface preparation procedures, and a clearly defined and reliable test

procedure are necessary for designers to be able to consider a material and predict its usefulness.

This program was initiated by the NASA Langley Research Center to investigate the relation between emittance and the method of preparing the surface for three oxidized metal surfaces and to point out clearly the variation in the results obtained with changes in surface-preparation procedures. The three metals were inconel, Inconel-X, and type 347 stainless steel. Four basic types of surface-preparation procedures were used prior to oxidation; the resulting surface conditions are denoted as "as received", "etched", "grit-blasted", and "polished". These preoxidation treatments are described in detail in tables I and II. Other surfaces were formed by combining or varying these treatments or by varying the oxidation time. Thus, correlation of emittance characteristics with surface-preparation procedure was obtained for a large range of surface-preparation procedures. Data were also obtained on the reproducibility of the oxidized surfaces. Each specimen was tested by measuring the spectral normal emittance from 1.0 to 15.0 microns at 900° F, 1,200° F, 1,500° F, and 1,800° F. Spectral rather than total emittance measurements were made in order to provide more insight into the effects of surface preparation on the emittances of these oxidized alloys. → 13

SYMBOLS AND DESIGNATIONS

$E_{\lambda,N}$	spectral normal emittance
V	recorder deflection
f	radiometer output, rms volts

Subscripts:

S	specimen
B	blackbody
0	denotes offset zero line for recorder

Designations (see tables I and II):

I	inconel
I-X	Inconel-X
S	type 347 stainless steel
AR	as received
E_1, E_2	etched
GB_1, GB_2	grit blasted

P polished

$O_{1,2,3,4, \text{ or } 5}$ oxidation procedure

APPARATUS

The apparatus under consideration can be divided into two main parts - a spectrophotometer with its detector and recorder and a furnace, which contains the test specimen and a reference blackbody cavity, and an associated temperature-monitoring system and power supply. These two parts of the apparatus are described in detail in the following sections.

Spectrophotometer and Associated Apparatus

A commercially available spectrophotometer was used in this investigation. Minor modification was made to the optical system to allow the use of an external source. These modifications together with the original optical path are shown in figure 1. Mirrors M_1 and M_2, as well as the internal source of the standard instrument, were removed. Mirrors M_3 (a 6-inch focal-length parabolic mirror) and M_4 (a plane-surface mirror) were positioned to transmit radiation from the external source and to focus this radiation at the same point as the original instrument. A new cover for this portion of the instrument was designed with an aperture and water-cooled shutter so that the radiation from the external source could be prevented from passing into the instrument, when so desired, and reradiation from the shutter could be prevented. A new base plate for this cover was designed so that the optics could be changed back to the standard optics in about 30 minutes.

The spectral region from 1.0 to 15.0 microns was chosen for this study since at 1,800° F, the maximum temperature, there is approximately 0.33 percent of the total energy below 1.0 micron and only 1.7-percent energy above 15.0 microns. At 900° F, the lowest temperature, there is approximately no energy below 1.0 micron and only 6.4-percent energy above 15.0 microns. A sodium chloride prism with a high-speed evacuated thermocouple detector with a potassium bromide window were chosen to cover this spectral region.

A 1/4-second-response strip-chart recorder is used to record the data.

Furnace and Associated Apparatus

The furnace shown in figure 2 is designed similarly to that used by McMahon (ref. 1) in his investigation of glasses. The principle involved in this type of furnace is that any body placed in a blackbody enclosure at thermal equilibrium will be at the temperature of the blackbody enclosure.

This furnace is constructed essentially in three cubes. The inner cube, called the liner, serves as the blackbody cavity. It is composed of heavily

oxidized inconel approximately 1/16 inch thick and 6 inches on each side. This liner fits inside a cubical shell of silicon carbide which serves as the furnace core. The silicon carbide is flame-sprayed with alumina on the outside to increase its strength and then wound with AWG 17-gage Nichrome V resistance wire that serves as the heating element. The core is well insulated by an outer cubical wall of high-temperature refractory firebrick. All these cubes are fabricated in two halves to facilitate placement of the specimen. The lower half of the furnace contains a blackened, water-cooled viewport which is tapered from 1/4-inch diameter on the inside to 3/8 inch on the outside of the furnace. A provision for rotating the test specimen within 1/16 inch of the inconel liner by an external 15-rpm motor is also in this half of the furnace.

Since the furnace is divided into two halves and the Nichrome V windings are separated, the power is supplied and controlled by two continuously variable 20-ampere autotransformers.

In operation, four AWG 30-gage chromel-alumel thermocouples are spot welded to the outer surface of the inconel liner. These thermocouples are positioned at the center of the top and bottom of the liner and on the center of the top right side and bottom left side as illustrated by the circles on the right side of figure 3. The thermocouples are constantly monitored to adjust and hold the temperature of the cavity (and thus the temperature of the test specimen) at the desired value.

CALIBRATION AND ERRORS

Spectrophotometer

Since several prisms are interchangeable for this spectrophotometer, the wavelength indicator is divided in arbitrary units. Each prism used in this instrument must, therefore, be calibrated through the wavelength region covered by the prism. The sodium chloride prism was calibrated from 1.0 to 15.0 microns by the use of absorption and emission spectra of known compounds and elements. A calibration curve of wavelength indicator number as a function of the wavelength in microns was then constructed.

Furnace

In order to determine whether any temperature differential exists between a semicircular test specimen and the cavity, an inconel specimen with three thermocouples attached, as shown on the left of figure 3, was placed in the furnace out of view of the port. The temperature of the cavity was measured by thermocouples attached at eight points on the inconel cavity. The placement of these eight thermocouples is shown on the right of figure 3.

The temperatures of the specimen and of the cavity were measured over the temperature range from 600° F to 1,800° F and were found to be in close agreement as shown in figure 4. Although these temperatures are averages of all

thermocouples on the specimen or cavity, the actual differences are less than 1.0 percent. The specimen temperature was lower than the cavity temperature over the entire temperature range, but the maximum deviation was only 0.87 percent of the absolute temperature. These measurements indicate that the assumption of thermal equilibrium between specimen and cavity is valid for the nonmeasuring portion of the specimen cycle. Thus, the measurement of cavity temperature may be taken also as specimen temperature with a possible total-emittance error of less than 3.5 percent. The spectral-emittance error for this possible temperature difference, as calculated by the method shown in appendix II of reference 2, at 600° F would vary from 11.0 percent at 1.0 micron to 1.20 percent at 15 microns; and at 1,800° F would vary from 3.50 percent at 1.0 micron to 0.40 percent at 15 microns.

An estimate of the emittance of the cavity, based on the results of calculations outlined in reference 3, gives a value of 0.999; hence the cavity is considered to be a blackbody.

When the specimen is rotated in front of the viewing port it radiates to the cooler surroundings and obviously must cool. The rate of cooling is determined by many factors, one of which is the emittance of the specimen. To determine the effects of this cooling on emittance measurements, the following procedure was used. An inconel specimen was placed in the furnace, out of sight of the viewport, and heated to 1,800° F. After the furnace temperature was stabilized, measurements of radiant flux intensity were obtained by use of a total radiation bolometer. With the specimen out of view of the port the bolometer was focused into the cavity, and a measurement of the blackbody flux intensity was recorded. The semicircular specimen was rotated at 15 rpm and measurements for the blackbody and specimen were taken alternately for 12 consecutive cycles. The results are shown in figure 5 where

f_S radiometer output for specimen, rms volts

f_B radiometer output for blackbody, rms volts

The gradual decrease of the ratio of the radiant flux intensities of specimen and blackbody is an indication of the cooling effect. The specimen emittance measured at 1/2 cycle (the first viewing cycle) was 0.95, and that measured at 3/2 cycles was 0.94. This decrease, about 1 percent, is considered to represent an upper limit of this cooling error, since the experiment was performed with a high emitting specimen at the highest furnace temperature. In practice, the specimen is rotated past the viewport only once (corresponding to the first viewing cycle on the figure) per reading, and is allowed to heat again for 1 minute before making a reading at the next wavelength; accordingly, this error is probably appreciably below this estimated upper limit.

The reproducibility of emittance values using this system, as measured during this study, is within 0.5 percent. An example of the reproducibility of the measurements is shown in figure 6.

The total error possible in considering all these factors will not exceed 5.0 percent for total normal emittance and 9.8 percent at 1.0 micron and 900° F to 1.9 percent at 15 microns and 1,800° F for spectral normal emittance.

MEASUREMENT PROCEDURES

Specimens used in this investigation were positioned on the rotating shaft in the furnace, normal to the axis of the viewport to pass within a maximum distance of 0.060 inch of the viewport for each cycle of rotation. By manual control of the autotransformers, the furnace is brought to equilibrium at the desired temperature.

An example of a typical recorder trace is shown in figure 7 for a particular temperature and wavelength. The recorder zero V_0 is displaced up scale by an additive signal to allow the complete monitoring of any change in this setting. The monochromator is then set at the desired wavelength and the water-cooled shutter is opened. The radiant flux intensities from the blackbody reference V_B and from the test specimen V_S are then recorded and the water-cooled shutter is closed. This procedure is repeated at 0.5-micron intervals from 1.0 to 15.0 microns. A large recorder deflection is obtained by attenuator adjustments for each reading to minimize the error in calculating emittance values from recorder charts. The calculation of the spectral normal emittance $E_{\lambda,N}$ is then obtained by the ratio:

$$E_{\lambda,N} = \frac{V_S - V_0}{V_B - V_0}$$

A computer program was established to calculate the total normal emittance from the spectral-normal-emittance values obtained from the previously outlined procedure.

The adherence of the oxide formed on these alloys was checked by the use of the cellulose transparent-tape test. This test was performed by pressing a piece of cellulose transparent tape on the surface of the specimen and pulling it off. Upon examination of the tape and the specimen, an indication was obtained for the relative adherence of the oxide to the specimen.

SPECIMENS

The test specimens used in this study were half of a 4-inch-diameter circular disk 1/16 inch thick. The nominal chemical composition of the alloys studied, inconel (ref. 4), Inconel-X (ref. 5), and type 347 stainless steel (ref. 6) is given in the following table:

Composition of inconel, percent	
Nickel	77.00
Chromium	15.00
Iron	7.00
Manganese	0.25
Copper	0.20
Silicon	0.25
Carbon	0.08
Sulfur	0.007

Composition of Inconel-X, percent	
Nickel (minimum)	70.00
Chromium	14.00 to 16.00
Titanium	2.25 to 2.75
Niobium	0.70 to 1.20
Aluminum	0.40 to 1.00
Iron	5.00 to 9.00
Manganese	0.30 to 1.00
Silicon (maximum)	0.50
Copper (maximum)	0.20
Carbon (maximum)	0.08
Sulfur (maximum)	0.01

Composition of type 347 stainless steel, percent	
Iron (minimum)	70
Nickel	9 to 13
Chromium	17 to 19
Carbon (maximum)	0.08
Manganese (maximum)	2.00
Silicon (maximum)	1.00
Niobium-Tantalum (minimum)	10 × carbon content

SPECIMEN PREPARATION

Several oxidized surfaces were prepared on each of the three materials; inconel, Inconel-X, and type 347 stainless steel. These oxidized alloys, whose surface preparations are designated and described in tables I to V, were as received (AR), etched (E_1) or (E_2), grit blasted (GB_1) or (GB_2), and polished (P). Other surfaces were formed by combining these preoxidation treatments or changing the oxidation time. The exact method of preparing these test specimens is explained in detail in the following sections.

Discussion and Description of Preparation Procedures

Certain standard procedures were used in preparing most of the specimens used in this study. The specimens were degreased by swabbing with cotton swabs using methyl alcohol. This process was repeated with reagent grade acetone and followed by a thorough rinse in deionized water. These specimens were then air dried. If water spots appeared on the specimens after drying the process was repeated. Procedures for specimens whose preparation did not include the standard degreasing process are thoroughly described in their respective sections. Rubber gloves were used while preparing these specimens and cotton gloves were used in handling the specimens between stages of specimen preparation to avoid contamination from body oils or fingerprints. All specimens were oxidized in a clean electric furnace preheated to the desired oxidation temperature. The specimens were oxidized for the time and at the temperature required to provide the oxide surface desired for testing.

An adherent, reproducible oxidized surface is normally easily obtainable on inconel and Inconel-X but is more difficult to obtain on the type 347 stainless steel. Care must be taken to maintain the oxidation temperature of the stainless steel at 1,800° F since the oxide becomes less adherent with an oxidation temperature above 1,900° F. Care should also be taken to cool the stainless-steel specimens slowly before removal from the oxidation furnace. Slower cooling will produce a darker and more adherent oxide than a direct removal of the specimen from the furnace. In this study, the type 347 stainless-steel specimens were placed in a preheated electric furnace at 1,800° F and allowed to oxidize for 20 minutes. The power was then turned off and the furnace temperature was allowed to drop to about 900° F (approximately 30 minutes) before the specimen was removed. This procedure was used on all type 347 stainless-steel specimens.

The oxidation temperature and time for inconel and Inconel-X was 2,000° F for 20 minutes (listed as O_1 in table II) and for 347 stainless steel was 1,800° F for 20 minutes (listed as O_2 in table II) in all cases except where noted otherwise.

As Received

Those surfaces designated AR were given only the degreasing treatment before being oxidized. Otherwise, they were essentially as received from the supplier, no special handling was used in shipment but the materials used for testing had no gross scratches or irregularities.

Etched

Surfaces designated E were, after degreasing, placed in an etching solution. The specimens designated E_1 (only inconel and Inconel-X) were etched by a solution composed of 32 parts reagent grade nitric acid, 70-percent HNO_3, 8 parts reagent grade hydrofluoric acid, 48-percent HF, and 32 parts deionized water. The etching time was $1\frac{1}{2}$ hours. Upon removal from the solution the specimens were rinsed with deionized water, swabbed with methyl alcohol, rinsed with

deionized water again, and air dried. These specimens were oxidized at 2,000° F for a period of 20 minutes (O_1).

The specimens designated E_2 (only stainless steel) were etched by a solution composed of 100 ml of reagent grade hydrochloric acid, 36.5- to 38.0-percent HCL, 25 grams of powdered reagent grade chromic acid, 100-percent CrO_3, and 50 ml of deionized water. The etching time varied for this particular etching solution. A period of 5 minutes was allowed for the first two specimens and the time was decreased to 3 minutes as more specimens were etched in the same solution. (It was found that the action of the solution upon the surface of the stainless steel became more rapid with use.) The etched specimen was removed from the etching solution and immediately immersed in deionized water to rinse the residue left by the etching solution. The specimens were then swabbed with cotton in hot deionized water, then swabbed with methyl alcohol, and rinsed with deionized water and air dried. These specimens were oxidized at 1,800° F for a period of 20 minutes (O_2).

Polished

Surfaces designated P were degreased by the standard procedure and then hand polished through four papers (No. 240, 320, 400, and 600 grit papers). This process gives a flat, smooth surface, although it is not highly polished. The specimens were again cleaned by the standard procedure to remove all contaminants from the polishing process and finally oxidized as previously described.

Grit Blasted

Surfaces designated GB were first degreased in a vapor degreaser with trichloroethylene as the solvent. This was done to remove oils and other surface contaminants before blasting the surface. The specimens designated GB_1 were blasted with fresh No. 100 silicon-carbide grit under 90 psi at a distance of 6 inches. They were then again cleansed with the vapor degreaser to remove all fingerprints and contamination from the blasting process and were then air dried.

The surface GB_1 was prepared on all three alloys: inconel, Inconel-X, and type 347 stainless steel. The inconel and Inconel-X specimens were oxidized at 2,000° F for 20 minutes (O_1) while the stainless steel was oxidized for 20 minutes at 1,800° F (O_2).

The specimens designated GB_2 were prepared in the same manner as the GB_1 specimens except that the blasting process was done with fresh number 40-60 silicon-carbide grit under 90 psi at a distance of 6 inches.

This surface (GB_2) was also prepared on all three alloys. The stainless steel was oxidized at 1,800° F for 20 minutes (O_2). The Inconel-X was oxidized only at 2,000° F for 20 minutes (O_1), but the inconel was oxidized at 2,000° F for periods of 20 minutes (O_1), 2 hours (O_3), 4 hours (O_4), and 8 hours (O_5).

Other Surfaces

Other oxidized surfaces were formed by combinations of the preoxidation treatments. For these surfaces the oxidation time for the inconel and Inconel-X was 20 minutes at 2,000° F and for the 347 stainless steel was 20 minutes at 1,800° F. All combinations and the order of performing these treatments on the respective materials are listed in tables III to V.

RESULTS AND DISCUSSION

Inconel

The inconel specimens tested in this study are listed in table III with the preoxidation treatments, oxidation times and temperatures, the total normal emittances at 900°, 1,200°, 1,500°, and 1,800° F, and remarks on the physical conditions of the oxide surfaces. Six preoxidation treatments were used in investigating this alloy. These were AR, E_1, GB_2, P, GB_2 followed by E_1, and GB_1 followed by E_1. The spectral normal emittance from 1.0 to 15.0 microns is shown for the first four of these surfaces in figures 8 to 11 for temperatures of 900°, 1,200°, 1,500°, and 1,800° F. These curves present the data and show that the emittance increases with increasing temperature for every specimen. Other surfaces were formed by oxidizing GB_2 for four different time periods other than the standard 20-minute oxidation time.

The spectral normal emittance measured for the AR, E_1, GB_2, and P specimens is plotted in figures 12 and 13 for 900° F and 1,800° F, respectively. This allowed the effects of etching, grit blasting, or polishing to be compared with the as-received specimens.

Figures 12 and 13 show that the shapes of the spectral-normal-emittance curves for the E_1 and AR specimens are fairly similar. The curves for the GB_2 specimen were the highest and progressively increased in emittance with an increase in wavelength. The P specimen deviated from all others in this group by the fact that it tended to decrease in emittance with increasing wavelength and exhibited large maxima and minima. All specimens tended to increase in emittance with increasing temperature as shown from the total-normal-emittance values in table III.

Figures 14 and 15 for 900° and 1,800° F, respectively, are plots of spectral normal emittance for E_1, GB_2E_1, and GB_1E_1 which show the effects of grit blasting on the etched specimens and also compares the effects of the two grits being used in the blasting process before etching.

The shapes of the spectral-normal-emittance curves for E_1 and GB_1E_1 (figs. 14 and 15) are almost identical, with the curve for the E_1 specimen being slightly higher. The curve for the GB_2E_1 specimen is the most irregular, with two strong maxima. A visual comparison of the surface roughnesses of the E_1

and GB_1E_1 specimens showed that the E_1 specimen is definitely rougher than the GB_1E_1 specimen. The etching tended to remove the peaks from the blasting process and leave a very smooth surface on the GB_1E_1 specimen. All specimens tended to increase in emittance, as shown in table III, with an increase in temperature.

The standard oxidation time used in this study was 20 minutes. To investigate the change in the spectral normal emittance with change in the oxidation time at the same temperature (2,000° F), three GB_2 specimens were oxidized for longer periods of time - 2, 4, and 8 hours. These are designated at GB_2O_3, GB_2O_4, and GB_2O_5, respectively. The curves of spectral normal emittance are shown for these specimens and the GB_2O_1 specimen in figures 16 and 17 for 900° and 1,800° F, respectively.

The curves for the GB_2O_1 specimen lie appreciably below the curves for the specimens with longer oxidation times, and it also has a different shape. The curves for the GB_2O_3, GB_2O_4, and GB_2O_5 specimens were almost identical. These specimens increased in emittance with increases in oxidation time, as shown in table III; however, there was no appreciable increase in emittance after 2 hours. The GB_2O_3, GB_2O_4, and GB_2O_5 specimens in the 900° F tests increase in emittance to 3 microns and then are almost constant (except for the minimum at 6 microns) out to 14 microns. The 1,800° F tests on these three specimens show the emittance to be extremely high from 1 to 6 microns, beyond which they decrease and remain nearly constant (at about 0.95) to 15 microns.

Inconel-X

The Inconel-X specimens tested in this study are listed in table IV together with the preoxidation treatments, oxidation time and temperature, the total normal emittances for 900°, 1,200°, 1,500°, and 1,800° F, and remarks on the physical conditions of the oxide surfaces. Six preoxidation treatments were used in studying this alloy. These were AR, E_1, GB_2, P, GB_1 followed by E_1, and GB_2 followed by E_1. The spectral normal emittances from 1.0 to 15.0 microns are shown for the first four of these surfaces in figures 18 to 21 for temperatures of 900°, 1,200°, 1,500°, and 1,800° F. These curves present the data and show that the emittance increases with increasing temperature for every specimen.

Spectral-normal-emittance curves for the first four of these oxide surfaces are compared in figures 22 and 23 for 900° and 1,800° F, respectively. The curves are very similar, except that the curves for the polished specimen (P) develop a series of pronounced maxima and minima at wavelengths greater than 6 microns. The emittances of all these specimens increase with temperature, as shown by the total-normal-emittance values in table IV.

Figures 24 and 25 for 900° and 1,800° F, respectively, compare the spectral-normal-emittance curves for the E_1, GB_2, GB_2E_1, and GB_1E_1 specimens in order to show the effects of etching on the grit-blasted specimens.

The shapes of the spectral-normal-emittance curves of the GB_2, GB_2E_1, and GB_1E_1 specimens are very similar, but the curve for the etched specimen (E_1) lies generally below the others and has pronounced irregularities beyond about 7 microns. The emittances of these specimens increase with temperature, and the curves become less irregular in shape at the higher temperature. The curves for the grit-blasted specimens show that the GB_2 specimen was the highest emitter with GB_2E_1 next and GB_1E_1 the lowest.

Type 347 Stainless Steel

The type 347 stainless-steel specimens tested in this study are listed in table V together with the preoxidation treatments, oxidation time and temperature, the total normal emittances at 900°, 1,200°, 1,500°, and 1,800° F, and remarks on the physical conditions of the oxide surfaces. Six preoxidation treatments were used in the investigation of this alloy. These were AR, E_2, GB_2, P, GB_2 followed by E_2, and GB_1 followed by E_2. The spectral normal emittances from 1.0 to 15.0 microns are shown for the first four of these surfaces in figures 26 to 29 for temperatures of 900°, 1,200°, 1,500°, and 1,800° F. These curves present the data and show that the emittance increases with increasing temperature for every specimen.

Figures 30 and 31 for 900° and 1,800° F, respectively, compare the spectral normal emittance of the AR, E_2, GB_2, and P specimens. The curves for the AR, E_2, and GB_2 specimens have similar shapes, while the curves for the polished specimen (P) deviate greatly from these by decreasing in emittance with an increase in wavelength. It should be pointed out that the grit-blasted stainless-steel (GB_2) oxidized surface was definitely not reproducible. The GB_2 curves shown in figures 30 and 31 are for one of the higher emitting samples. These specimens increase in emittance with an increase in temperature.

Curves for the E_2, GB_2, GB_2E_2, and GB_1E_2 specimens are shown in figures 32 and 33 for 900° and 1,800° F, respectively. These curves follow the expected trend with GB_2 having the highest emittance, GB_2E_2 next, and GB_1E_2 having the lowest emittance of the grit-blasted specimens. These specimens also increase in emittance with increase in temperature.

Special Tests

Figures 34, 35, and 36 provide information on the reproducibility of the spectral-normal-emittance curves for similarly prepared specimens. Figure 34, which compares two samples of grit-blasted inconel, shows a nearly perfect reproducibility. Figure 35, which compares two samples of etched stainless steel, shows a less perfect agreement; however, the average difference is less than 2 percent.

Most of these specimens follow the same spectral-normal-emittance curve as their duplicate specimens. The as-received (AR) oxidized inconel specimens, when examined visually, looked grey-green in color and were slightly blotched. Since the coloration was somewhat uneven, it was considered that the spectral-normal-emittance curves would be most likely to show large variations from one sample to another. However, comparison of two specimens showed an average difference of only about 1 percent. This result led to tests of two additional samples prepared in the same manner. The extremes of these tests are shown in figure 37. The excellent agreement leads to the conclusion that an investigator cannot judge the reproducibility of the infrared emittance spectrum by examining the specimen visually.

After the emittances were determined, adherence tests were performed by use of the cellulose transparent-tape test. This is a destructive test, so one of the sample surfaces not chosen for testing was used. The results of these tests are given in tables III, IV, and V. An oxide surface was considered to be adherent if, upon performance of the cellulose transparent-tape test, only a few small particles could be found on the tape and no noticeable damage was done to the specimen. If pieces of the oxide surface were pulled off by the tape and the portion of the surface that had been covered by the tape did not resemble the remainder of the specimen, then it was listed as a nonadherent oxide.

CONCLUSIONS

This investigation has shown significant effects of preoxidation treatments and oxidation times on the spectral normal emittances of oxidized inconel, Inconel-X, and type 347 stainless steel. In general, if a grit-blasted surface is etched before being oxidized, the final oxidized surface will have a lower emittance but will be more adherent and uniform. Of the two types of grit used in this study, the coarser grit provided the higher emittance. Polishing, even to the degree used in this investigation, provided the lowest emittance of all specimens tested; possibly a high degree of polish, such as an electropolished surface, would result in even lower emittance. In the one set of tests in which oxidation time was varied for inconel, increasing oxidation time increased the emittance; increasing the oxidation time beyond 2 hours, however, produced no further effect.

For inconel, the different preoxidation treatment and oxidation times resulted in total normal emittances ranging from 0.79 (for the polished surface) to 0.97 (for the grit-blasted surface with extended oxidation time) at 1,800° F. At 900° F, the values ranged from 0.64 to 0.88, and the spectral emittance curves indicate that at lower temperatures the variation would be even greater. Because the surface-preparation procedures used in this investigation are not absolutely standardized, it is recognized that some workers using these procedures may obtain somewhat different results. However, the primary objective of this study, which was to obtain reasonably accurate emittance measurements that would show the variation in spectral-normal and total-normal-emittance values with changes in surface-preparation procedures, has been achieved. It is therefore concluded

that for accurate thermal-radiation calculations, emittance values should be obtained from sources that specify the exact surface-preparation procedures.

Langley Research Center,
National Aeronautics and Space Administration,
Langley Station, Hampton, Va., February 10, 1964.

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3. Gouffé, André: "Corrections d'ouverture des corps-noirs artificiels compte tenu des diffusions multiples internes." Rev. d'Optique, vol. 24, nos. 1-3, Jan.-Mar. 1945, pp. 1-10.

4. Anon.: Engineering Properties of Inconel. Tech. Bulletin T-7, The Int. Nickel Co., Inc., Revised, Mar. 1950.

5. Anon.: Inconel "X" - A High Strength, High Temperature Alloy, Data and Information. Dev. and Res. Div., The Int. Nickel Co., Inc., Jan. 1949.

6. ASM Committee on Wrought Stainless Steels: Wrought Stainless Steels. Metals Handbook, Taylor Lyman, ed., A.S.M. (Cleveland, Ohio), c.1961, pp. 408-431.

TABLE I.- PREOXIDATION TREATMENTS

Surface designation	Surface preparation
AR	This surface is prepared on materials as received from the supplier. The only treatment is a thorough degreasing. The specimens are swabbed with methyl alcohol, with deionized water, with reagent grade acetone, and finally rinsed with deionized water. The specimens are air dried.
E_1	Degreased as for the AR specimens and then etched. The etching solution used was composed of 32 parts reagent grade nitric acid, 70-percent HNO_3, 8 parts reagent grade hydrofluoric acid, 48-percent HF, and 32 parts deionized water. The etching time was $1\frac{1}{2}$ hours. The specimens were rinsed with deionized water, swabbed with methyl alcohol using cotton swabs, rinsed with deionized water again, and air dried.
E_2	Same as E_1 except the etching solution was composed of 100 ml of reagent grade hydrochloric acid, 36.5- to 38.0-percent HCL, 25 grams of powdered reagent grade chromic acid, 100-percent CrO_3, and 50 ml of distilled water. The etching time was 3 to 5 minutes. The specimens were swabbed with cotton in hot deionized water, then swabbed with methyl alcohol, rinsed with deionized water, and air dried.
GB_1	The as-received stock was degreased with trichloroethylene in a vapor degreaser and then blasted with number 100 silicon carbide grit under 90 psi at a distance of 6 inches. The specimens were then degreased by the use of the vapor degreaser with trichloroethylene, and air dried.
GB_2	Prepared the same as GB_1 except that the stock was blasted with number 40-60 silicon carbide grit under 90 psi at a distance of 6 inches.
P	The specimens were degreased as for the AR specimens, and hand polished through four papers, 240, 320, 400, and 600 grit. They were degreased again in the same manner as the AR specimens and air dried.

TABLE II.- OXIDATION

Designation	Oxidation procedure		Remarks
	Time, min	Temperature, °F	
O_1	20	2,000	Used in preparing inconel and Inconel-X specimens
O_2	20	1,800	Used in preparing the type 347 stainless-steel specimens
O_3	120	2,000	Used for inconel only
O_4	240	2,000	Used for inconel only
O_5	480	2,000	Used for inconel only

TABLE III.- OXIDIZED INCONEL

Specimen (a)	Preoxidation treatment (a)	Total normal emittance, °F, at -				Other surface properties
		900	1,200	1,500	1,800	
$AR0_1$	AR	0.64	0.68	0.76	0.80	Fairly adherent; reproducible; grey with small green blotches
$E_1 0_1$	E_1	0.69	0.71	0.77	0.81	Very adherent; highly reproducible; grey-black
$P0_1$	P	0.64	0.70	0.76	0.79	Fairly adherent; not very reproducible; green-grey
$GB_2 0_1$	GB_2	0.77	0.80	0.85	0.87	Not very adherent or reproducible; grey-green
$GB_2 E_1 0_1$	GB_2 followed by E_1	0.72	0.74	0.79	0.81	Rough; very adherent; highly reproducible; grey-black
$GB_1 E_1 0_1$	GB_1 followed by E_1	0.68	0.72	0.78	0.80	Smooth; very adherent; highly reproducible; grey-black
$GB_2 0_3$	GB_2	0.86	0.94	0.96	0.97	Adherent; reproducible; more green than the $GB_2 0_1$
$GB_2 0_4$	GB_2	0.88	0.94	0.96	0.97	Adherent; reproducible; stable. The green color has disappeared leaving a grey-black oxide
$GB_2 0_5$	GB_2	0.87	0.94	0.96	0.97	Very similar to $GB_2 0_4$

[a]See tables I and II.

TABLE IV.- OXIDIZED INCONEL-X

Specimen (a)	Preoxidation treatment (a)	Total normal emittance, °F, at -				Other surface properties
		900	1,200	1,500	1,800	
ARO_1	AR	0.82	0.84	0.86	0.88	Adherent; uniform; reproducible; grey-black
E_1O_1	E_1	0.80	0.82	0.84	0.87	Very smooth; adherent; uniform; reproducible; grey-black
PO_1	P	0.80	0.82	0.86	0.89	Not very reproducible; light grey blotches
GB_2O_1	GB_2	0.85	0.88	0.90	0.92	Rough; adherent; reproducible; slightly blotched grey-black
$GB_1E_1O_1$	GB_1 followed by E_1	0.85	0.88	0.89	0.91	Very uniform; adherent; reproducible; grey-black
$GB_2E_1O_1$	GB_2 followed by E_1	0.86	0.88	0.90	0.92	Very uniform; adherent; reproducible; grey-black

[a]See tables I and II.

TABLE V.- OXIDIZED TYPE 347 STAINLESS STEEL

Specimen (a)	Preoxidation treatment (a)	Total normal emittance, °F, at -				Other surface properties
		900	1,200	1,500	1,800	
$AR0_2$	AR	0.82	0.86	0.89	0.91	Not very reproducible; nonuniform; blotched light grey
E_20_2	E_2	0.84	0.87	0.90	0.91	Rough; uniform; adherent; reproducible; grey-black
$P0_2$	P	0.74	0.77	0.81	0.83	Very smooth; uniform; adherent; reproducible; grey-black
GB_20_2	GB_2	0.85	0.88	0.90	0.92	Not reproducible; nonuniform; blotched
$GB_1E_20_2$	GB_1 followed by E_2	0.81	0.83	0.86	0.88	Smoother than E_20_2; uniform; adherent; reproducible: grey-black
$GB_2E_20_2$	GB_2 followed by E_2	0.84	0.86	0.88	0.90	Rougher than E_20_2; uniform; reproducible; adherence not as good as $GB_1E_20_2$; grey-black

[a]See tables I and II.

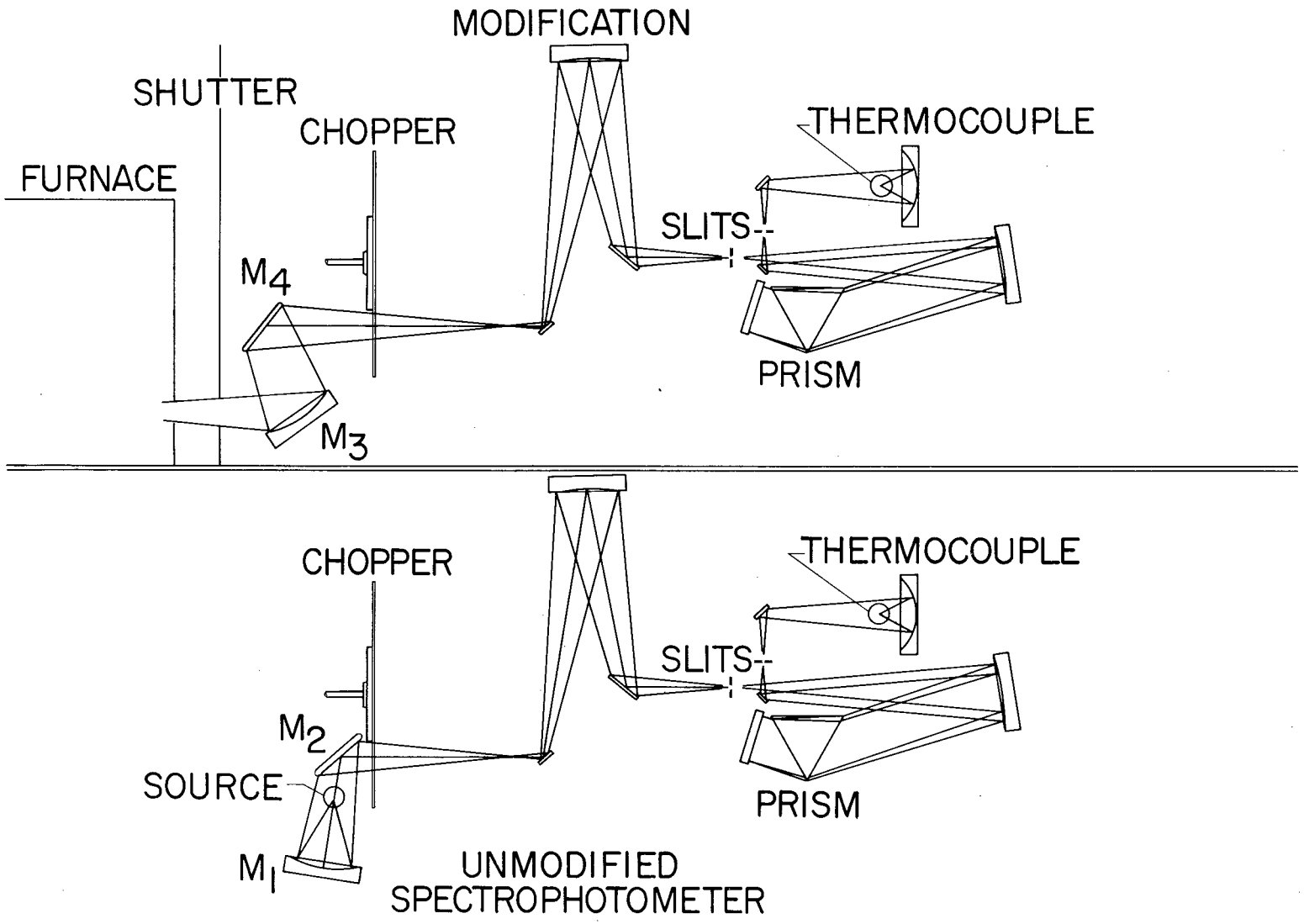

Figure 1.- Optical schematic of spectrophotometer.

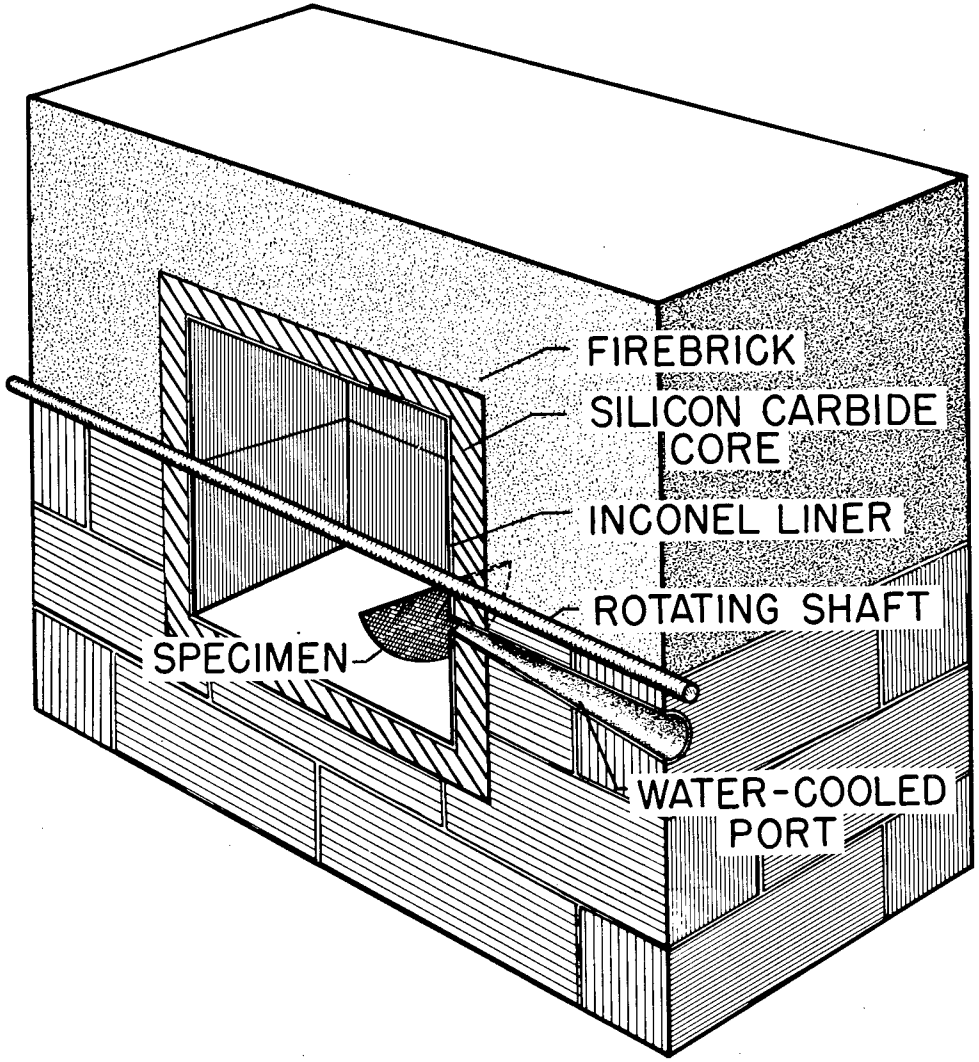

Figure 2.- Cross section of blackbody furnace.

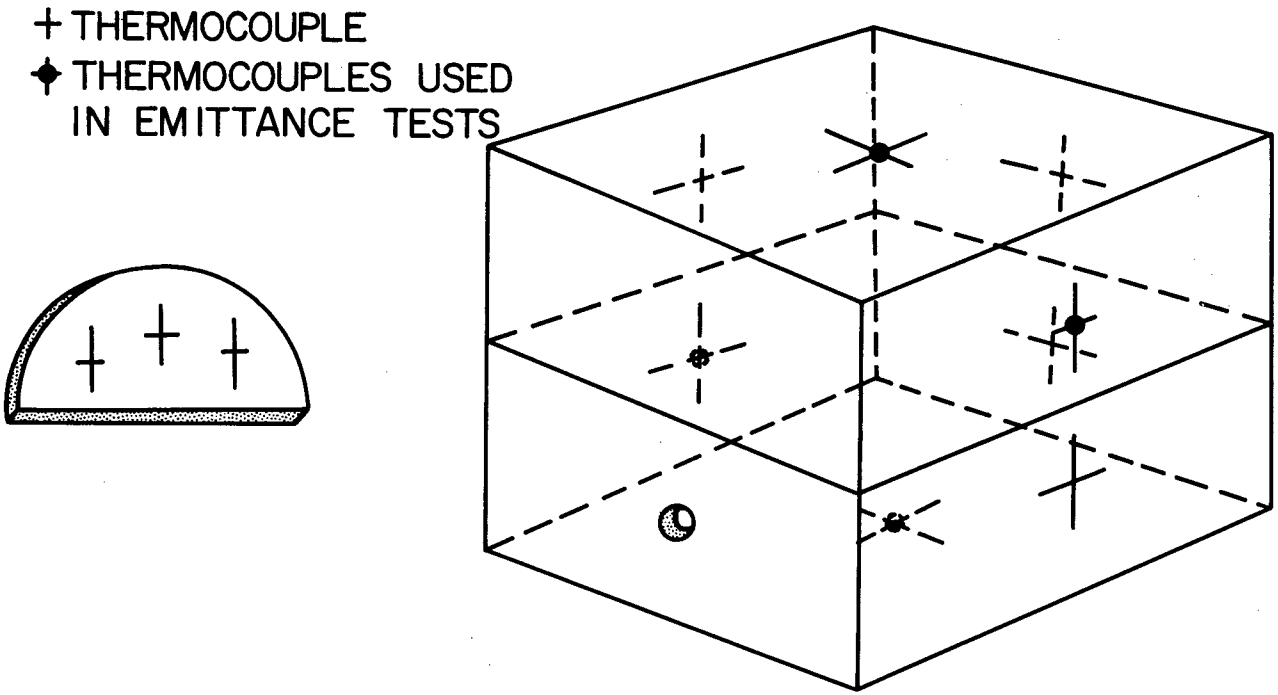

Figure 3.- Thermocouple placement on specimen and cavity.

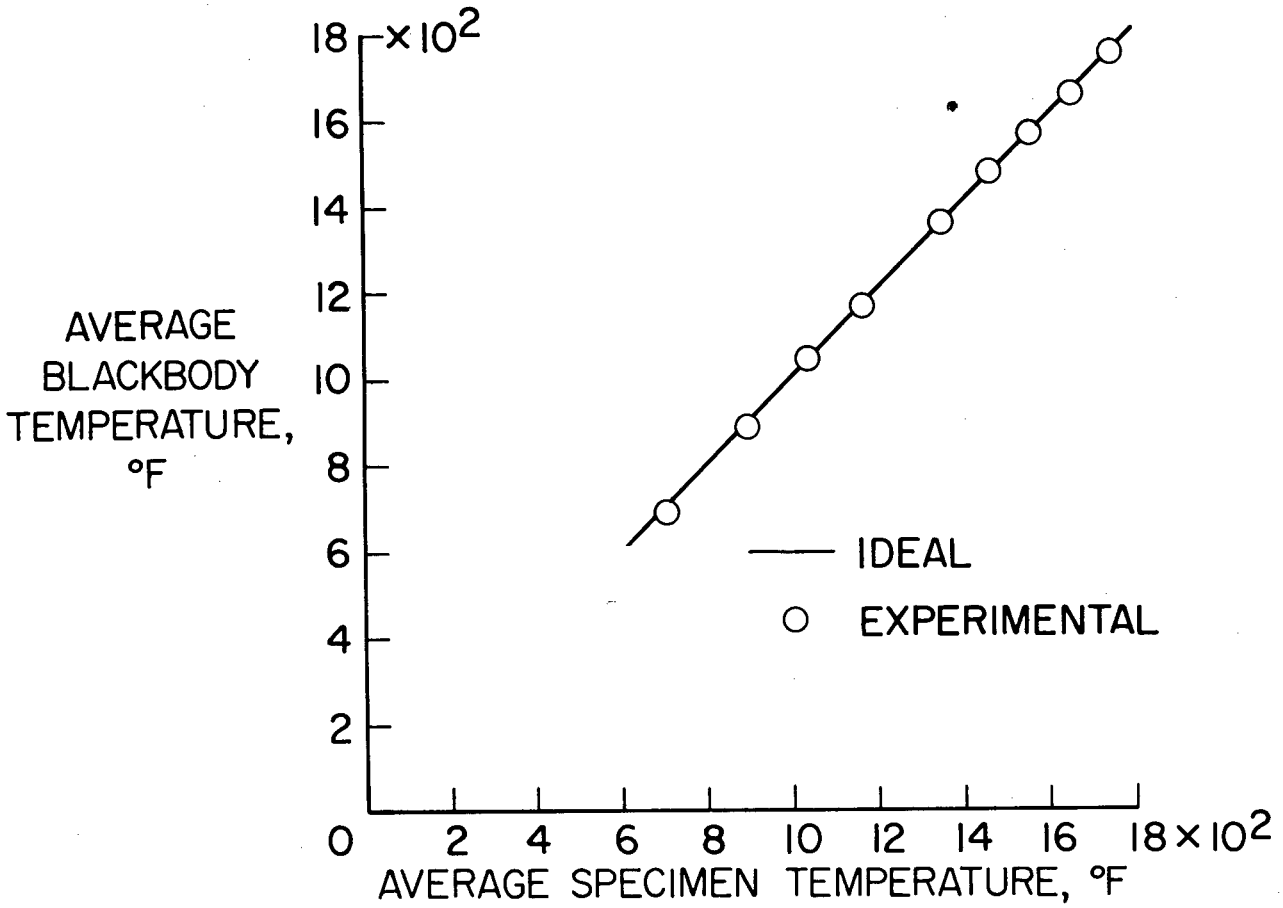

Figure 4.- Deviation of specimen temperature from blackbody temperature.

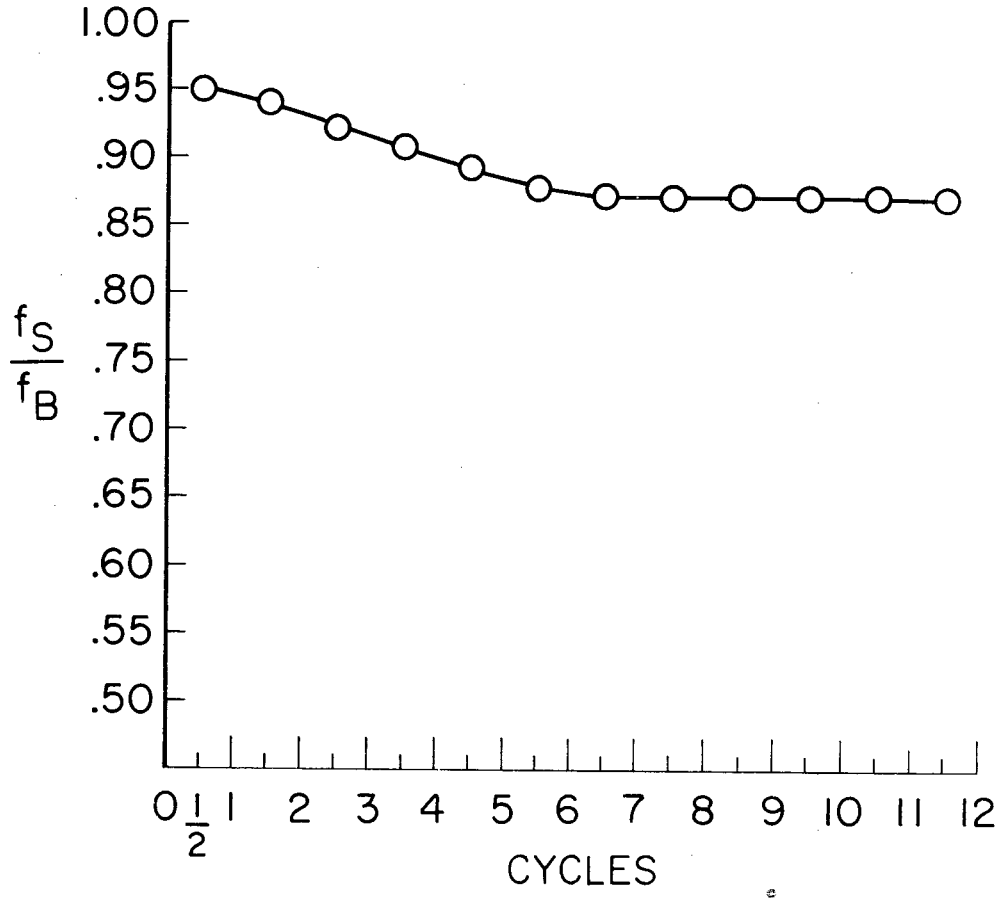

Figure 5.- Decrease of radiant intensity ratio for specimen of grit-blasted oxidized inconel at a furnace temperature of 1,800° F.

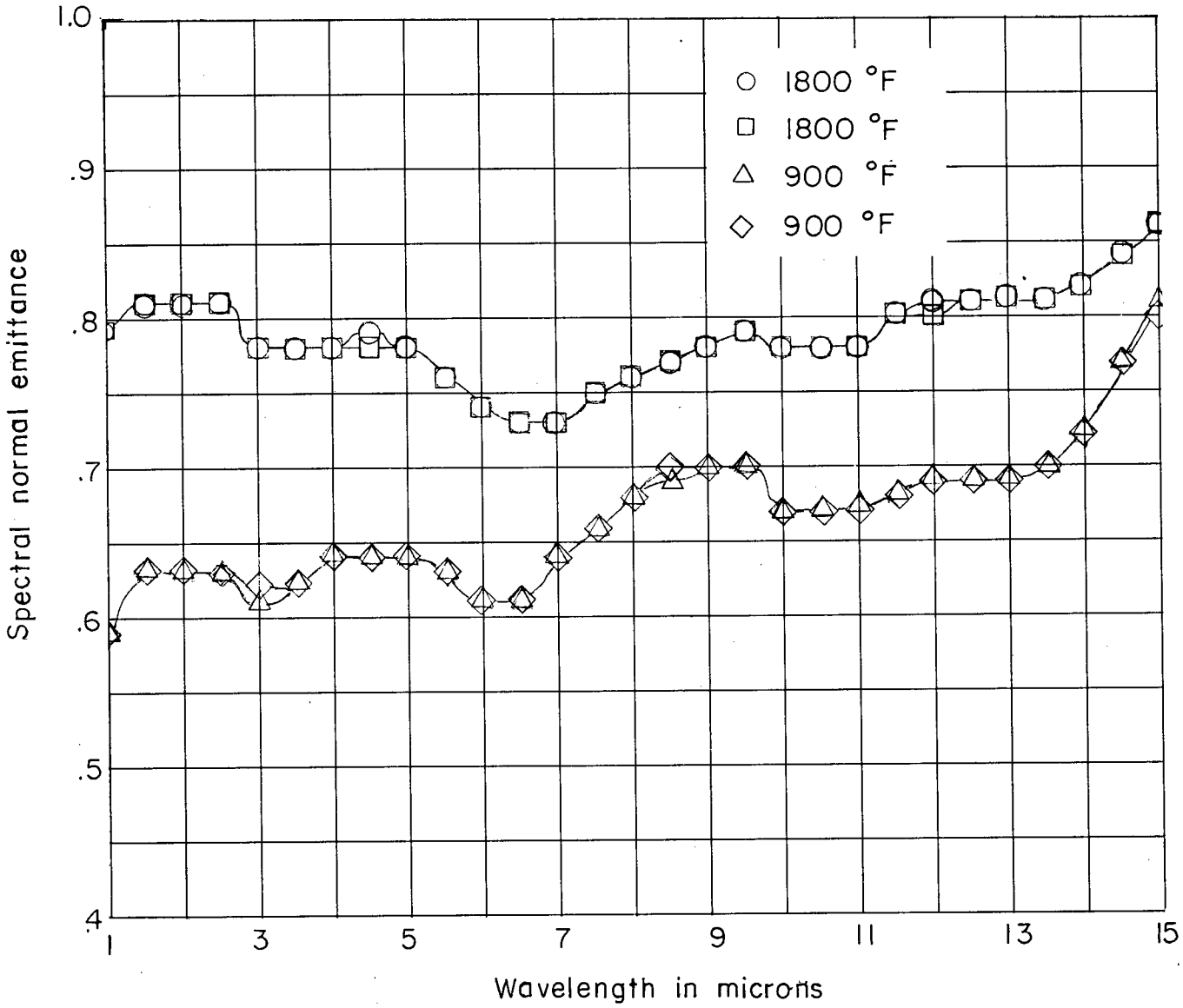

Figure 6.- Reproducibility of etched oxidized inconel (IE_1O_1) specimens.

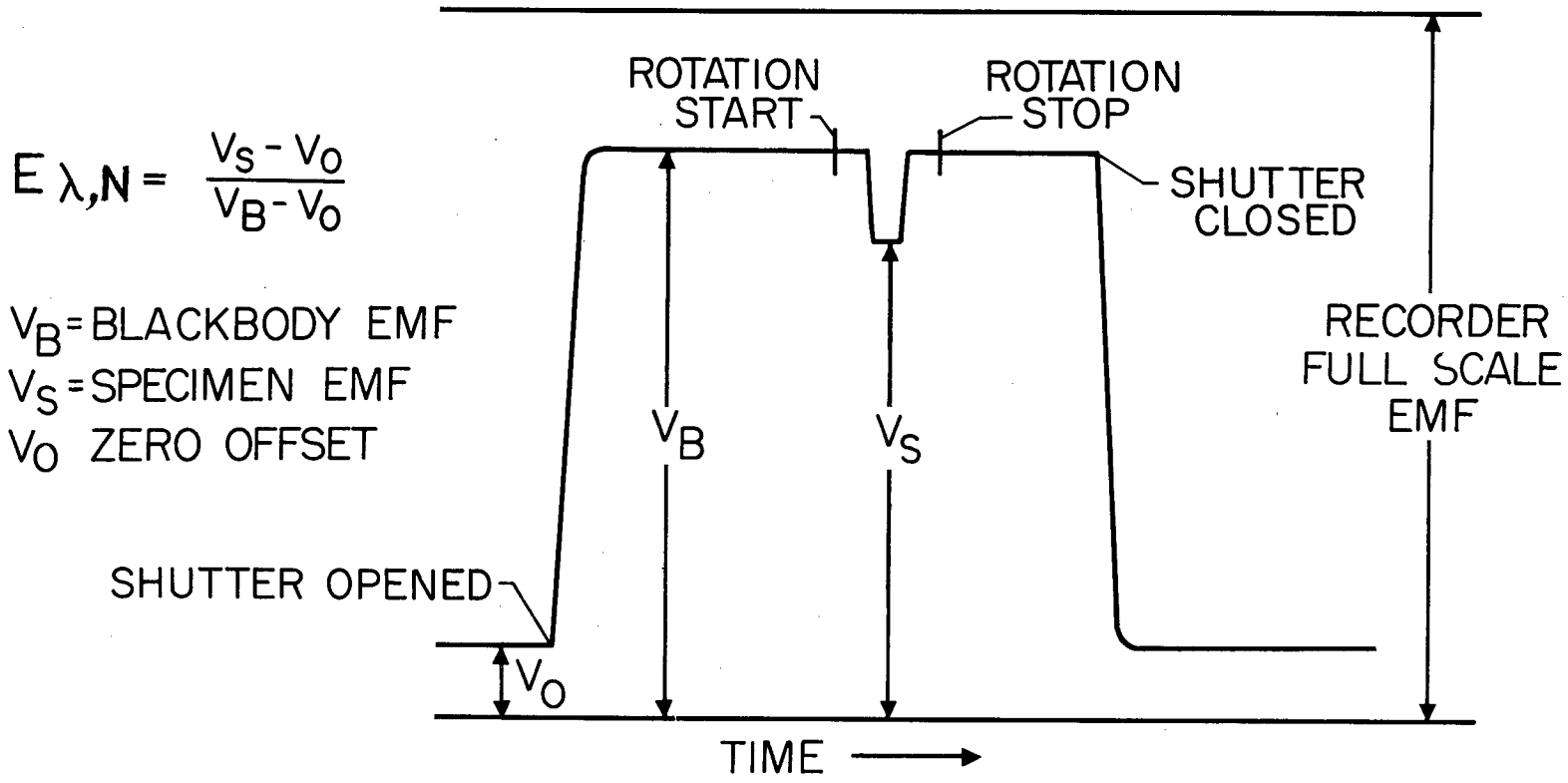

Figure 7.- Typical test record.

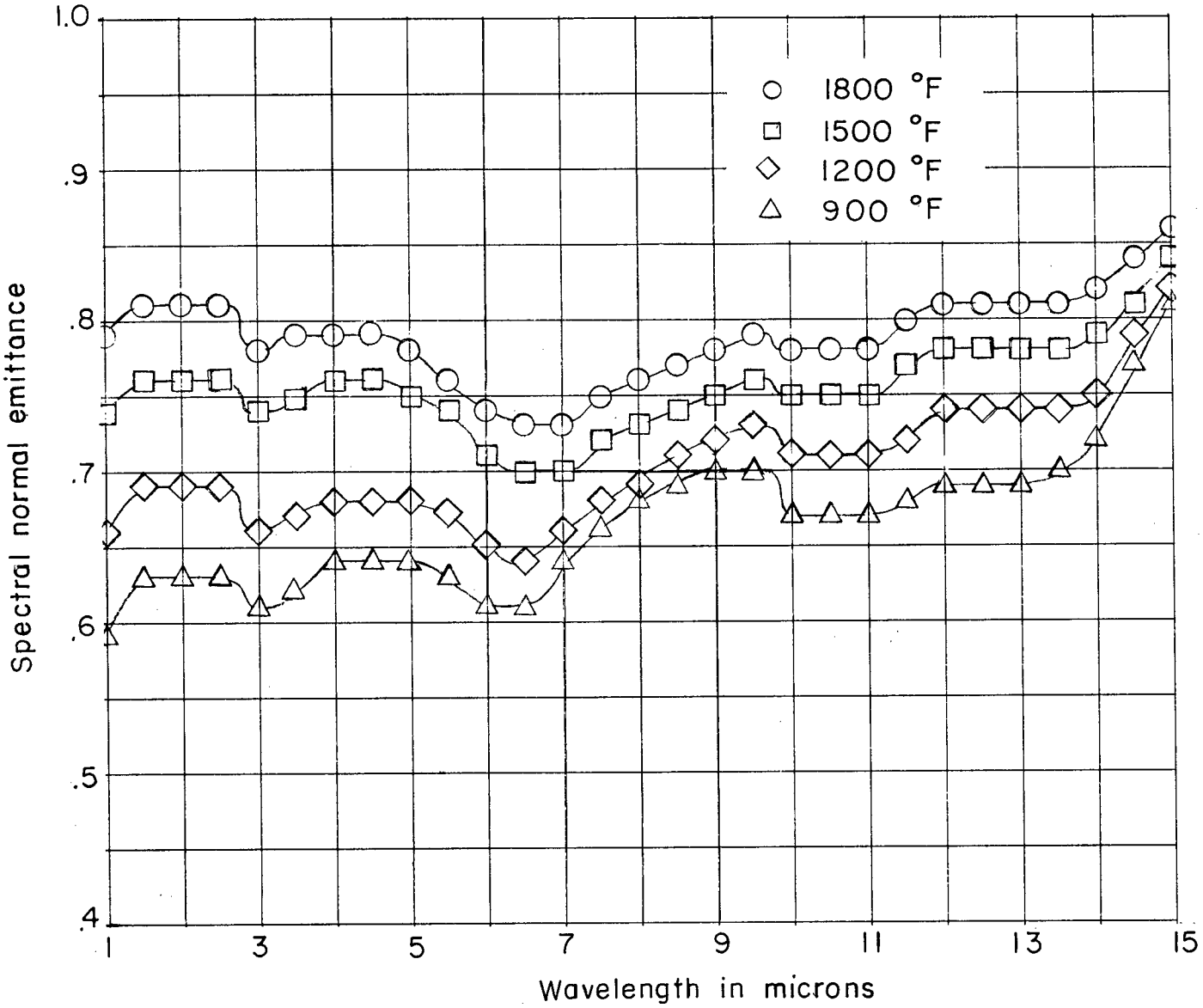

Figure 8.- Spectral normal emittance of oxidized as-received inconel ($IARO_1$) specimens.

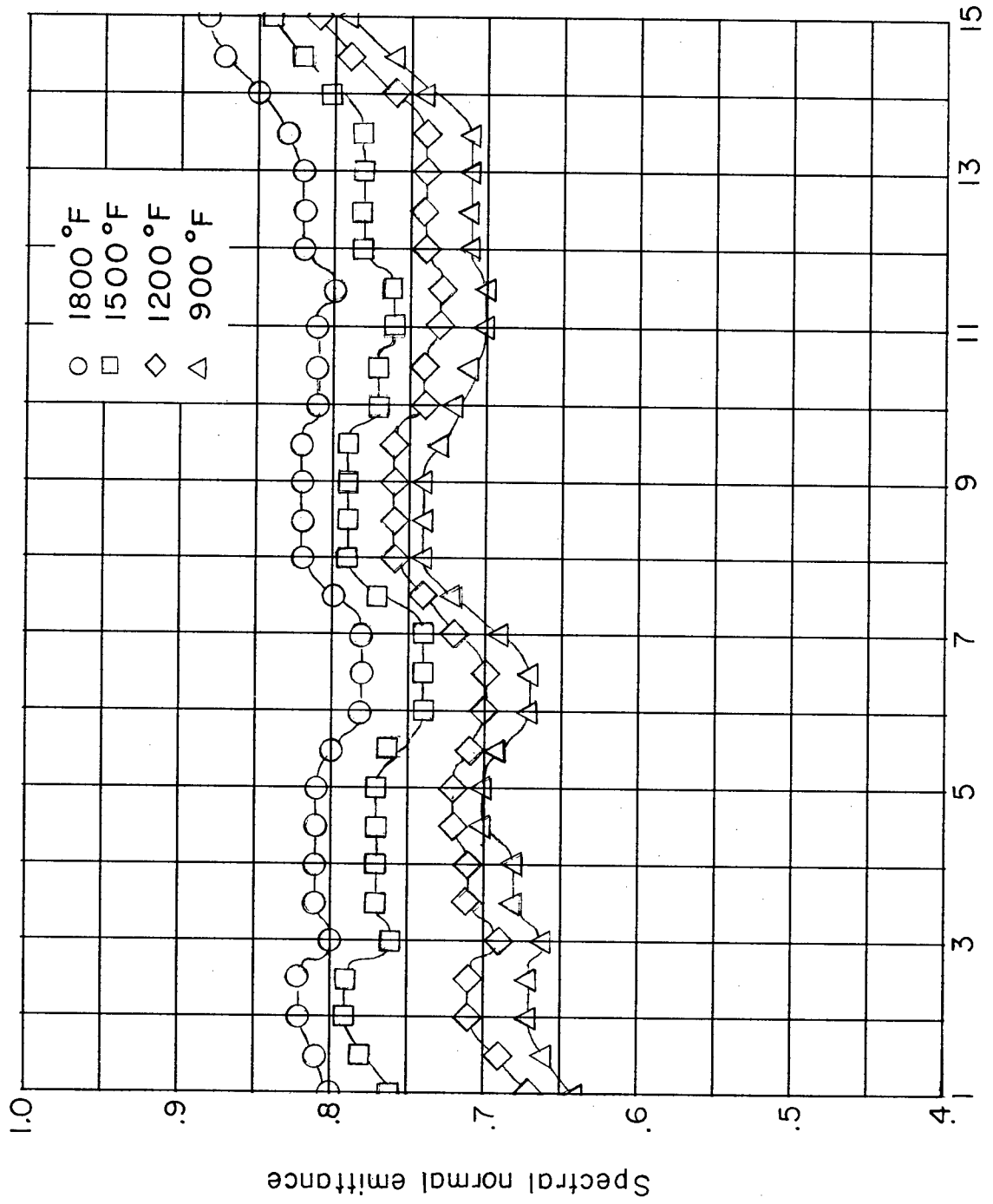

Figure 9.- Spectral normal emittance of oxidized etched inconel (IE_1O_1) specimens.

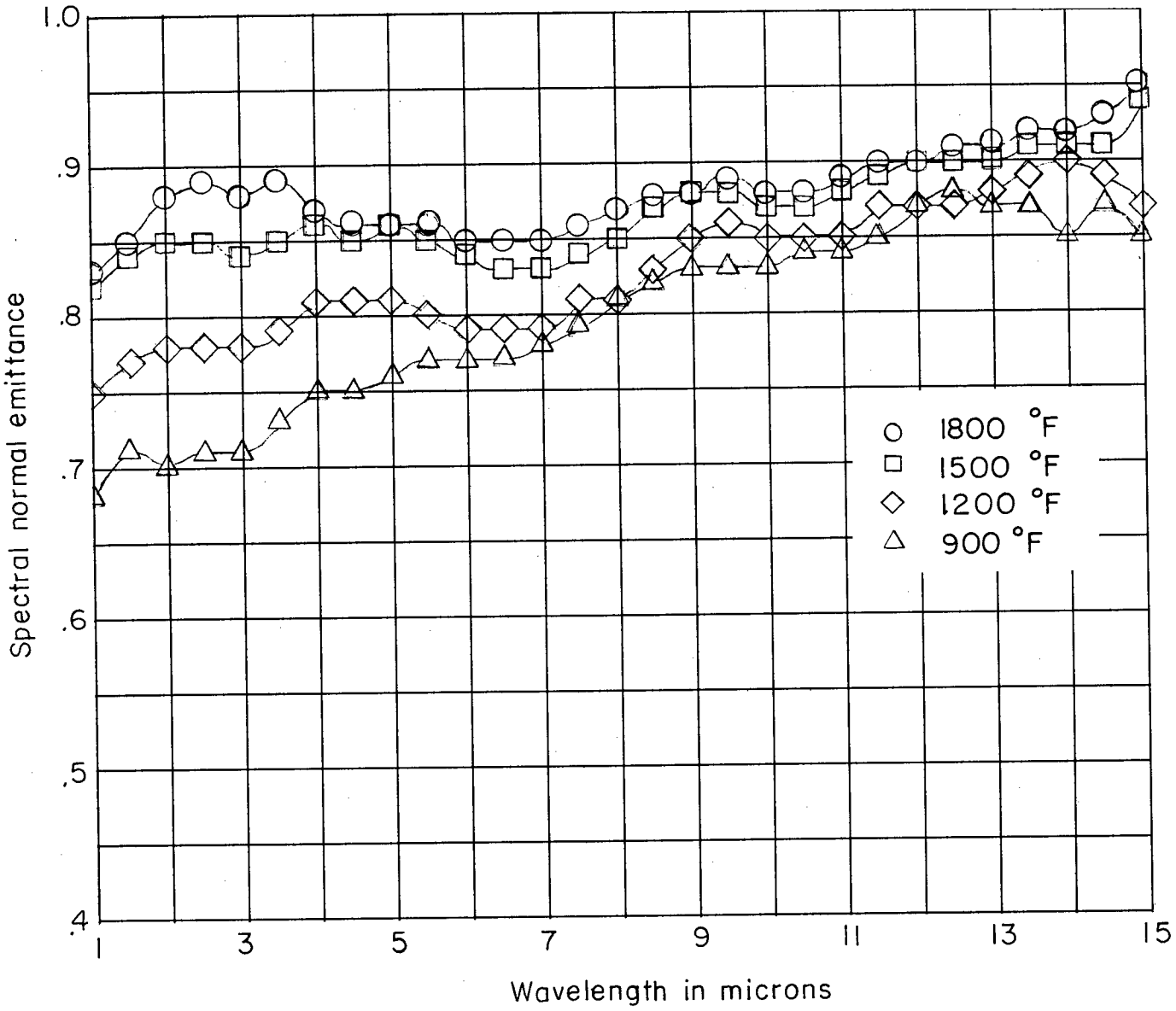

Figure 10.- Spectral normal emittance of oxidized grit-blasted inconel (IGB_2O_1) specimens.

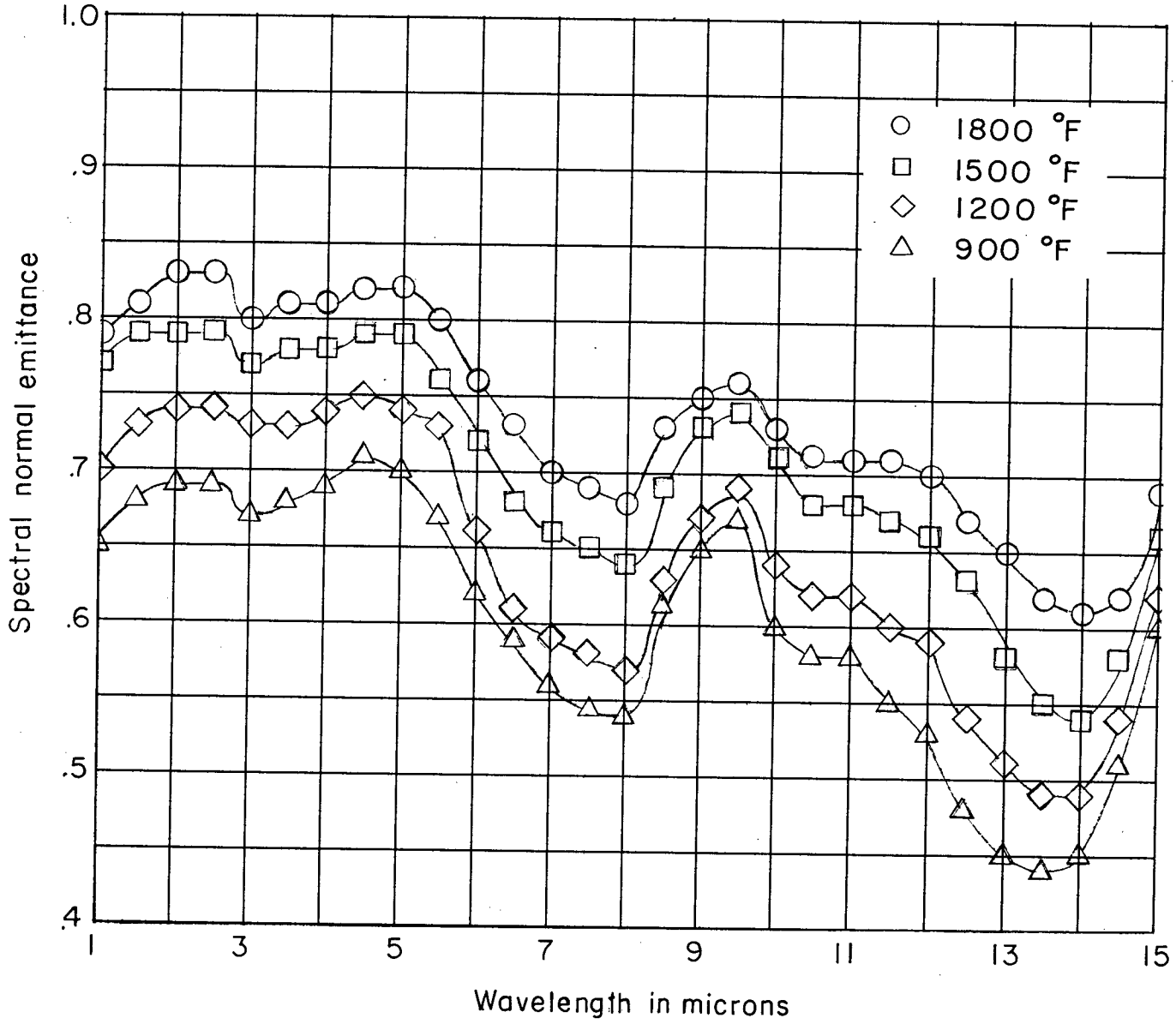

Figure 11.- Spectral normal emittance of oxidized polished inconel (IPO1) specimens.

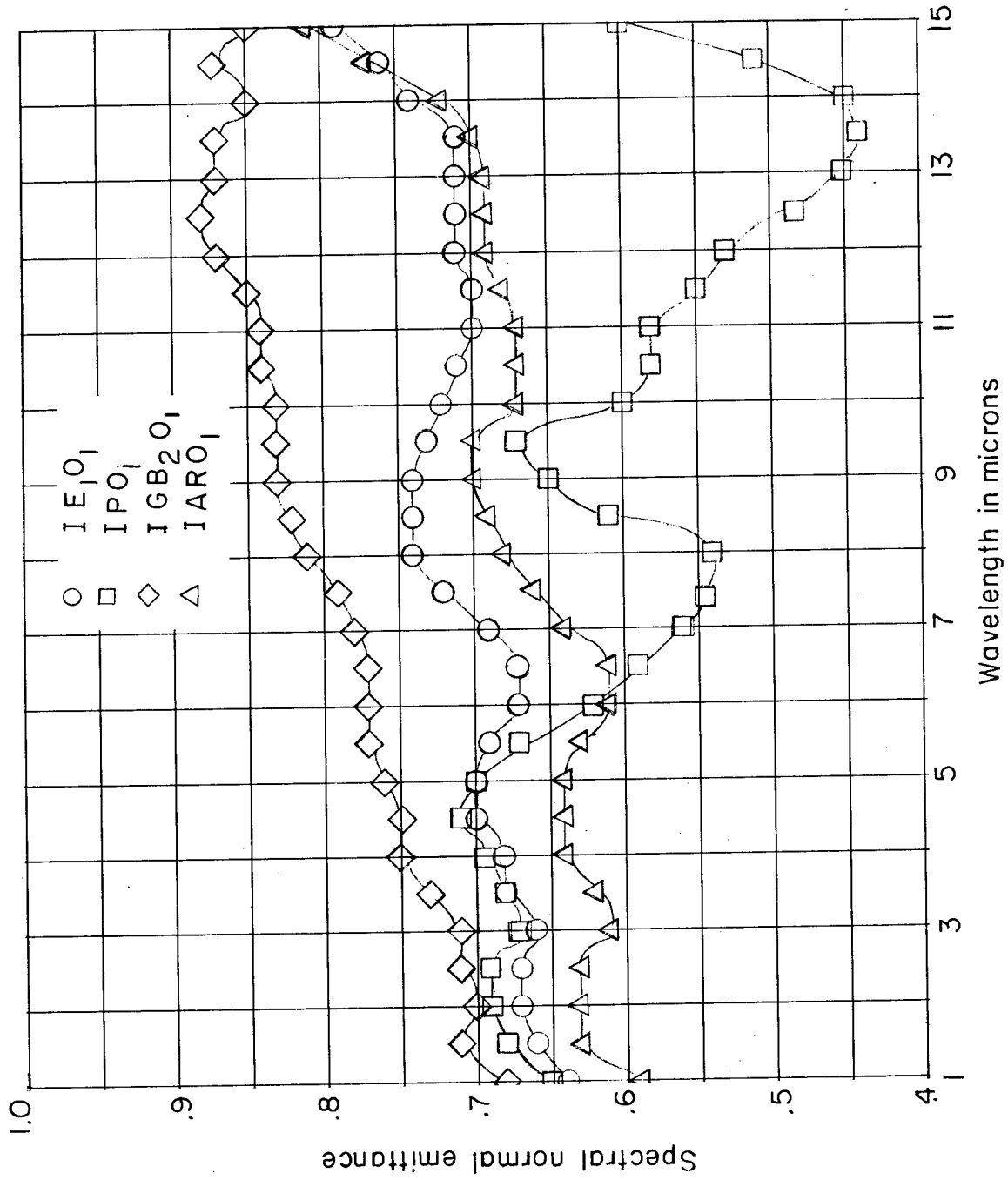

Figure 12.- Comparison of effects of preoxidation treatments on spectral normal emittance of oxidized inconel specimens at 900° F.

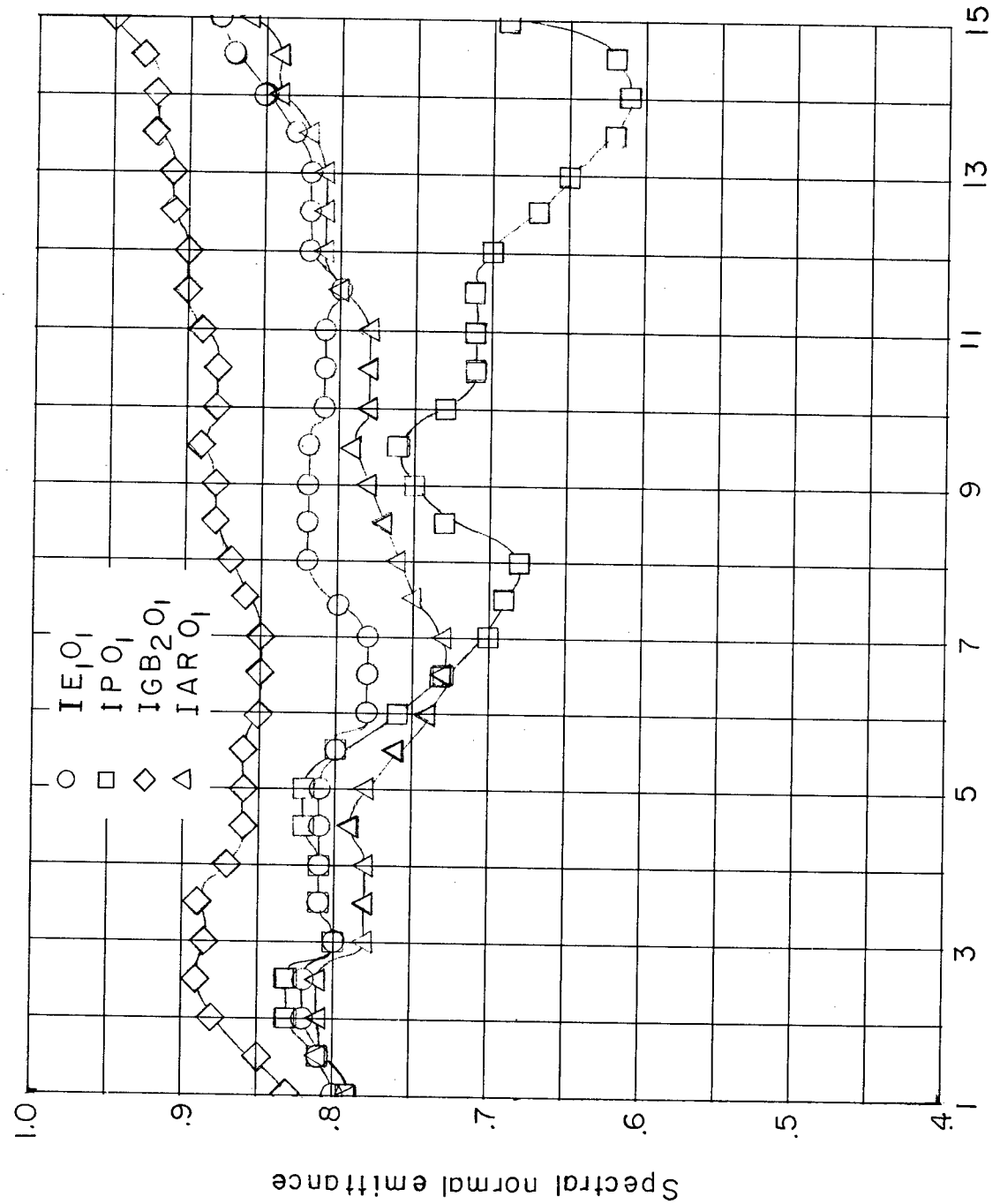

Figure 13.- Comparison of effects of preoxidation treatments on spectral normal emittance of oxidized inconel specimens at 1,800° F.

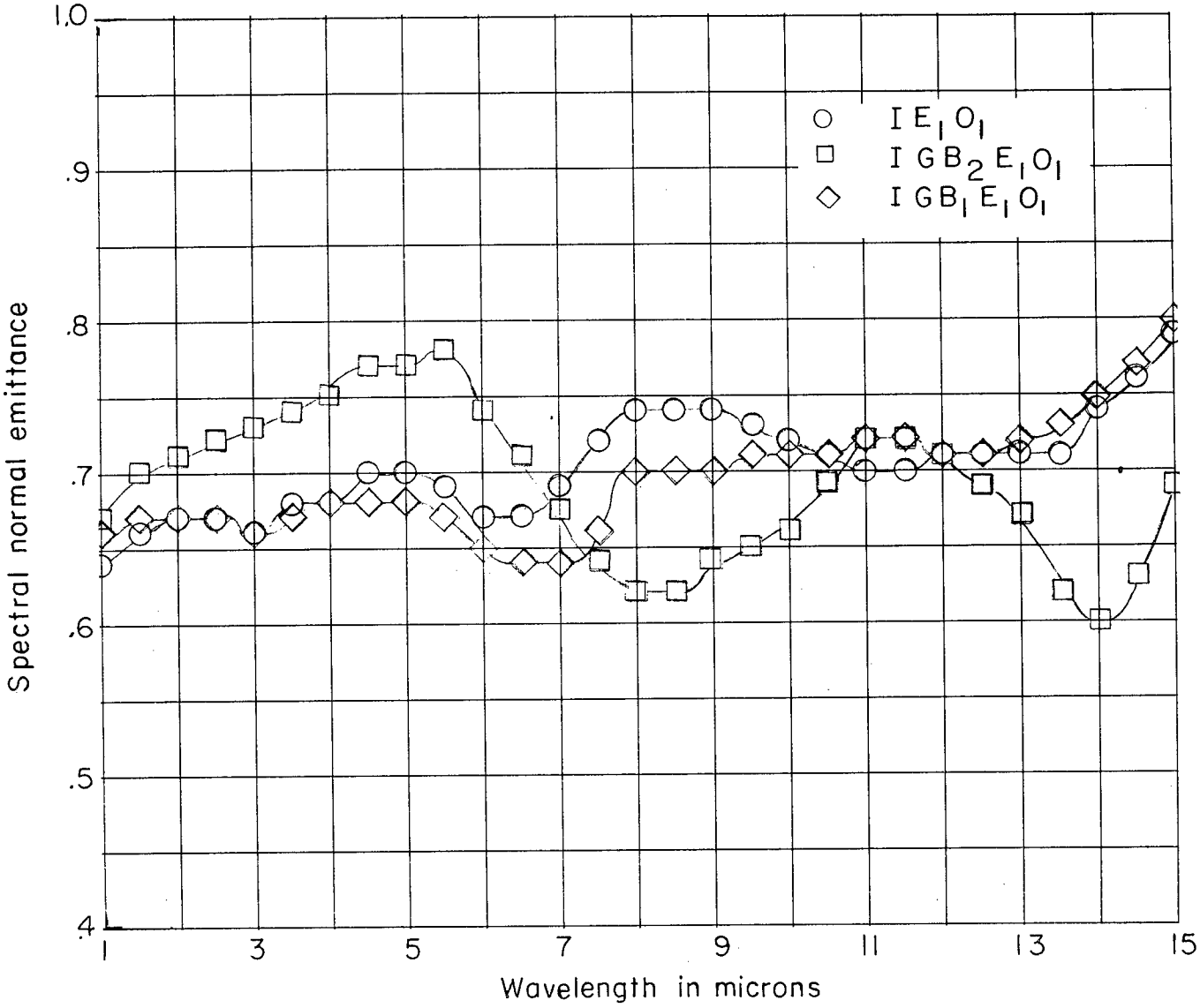

Figure 14.- Comparison of effects of combinations of preoxidation treatments on spectral normal emittance of oxidized inconel specimens at 900° F.

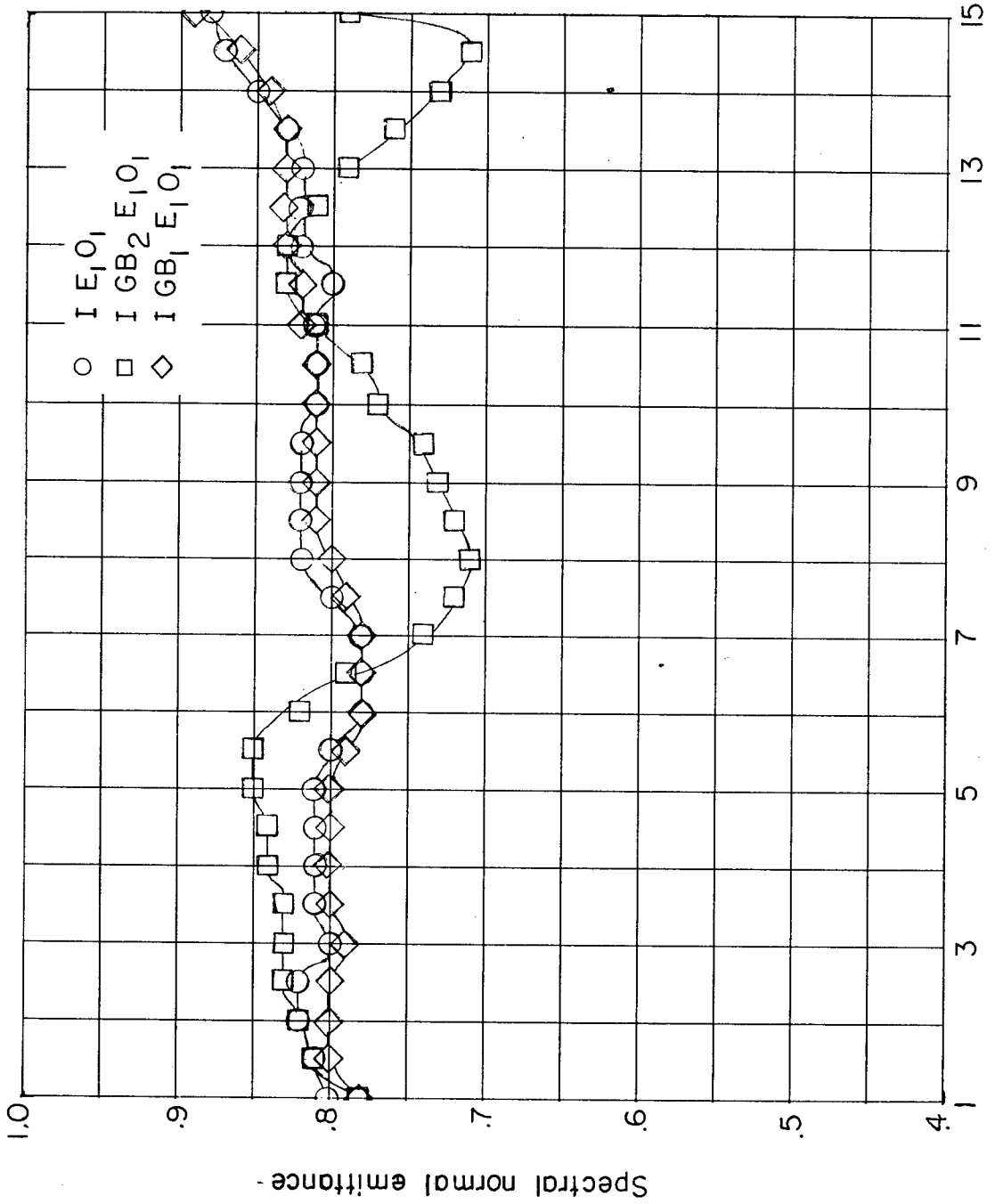

Figure 15.- Comparison of effects of combinations of preoxidation treatments on spectral normal emittance of oxidized inconel specimens at 1,800° F.

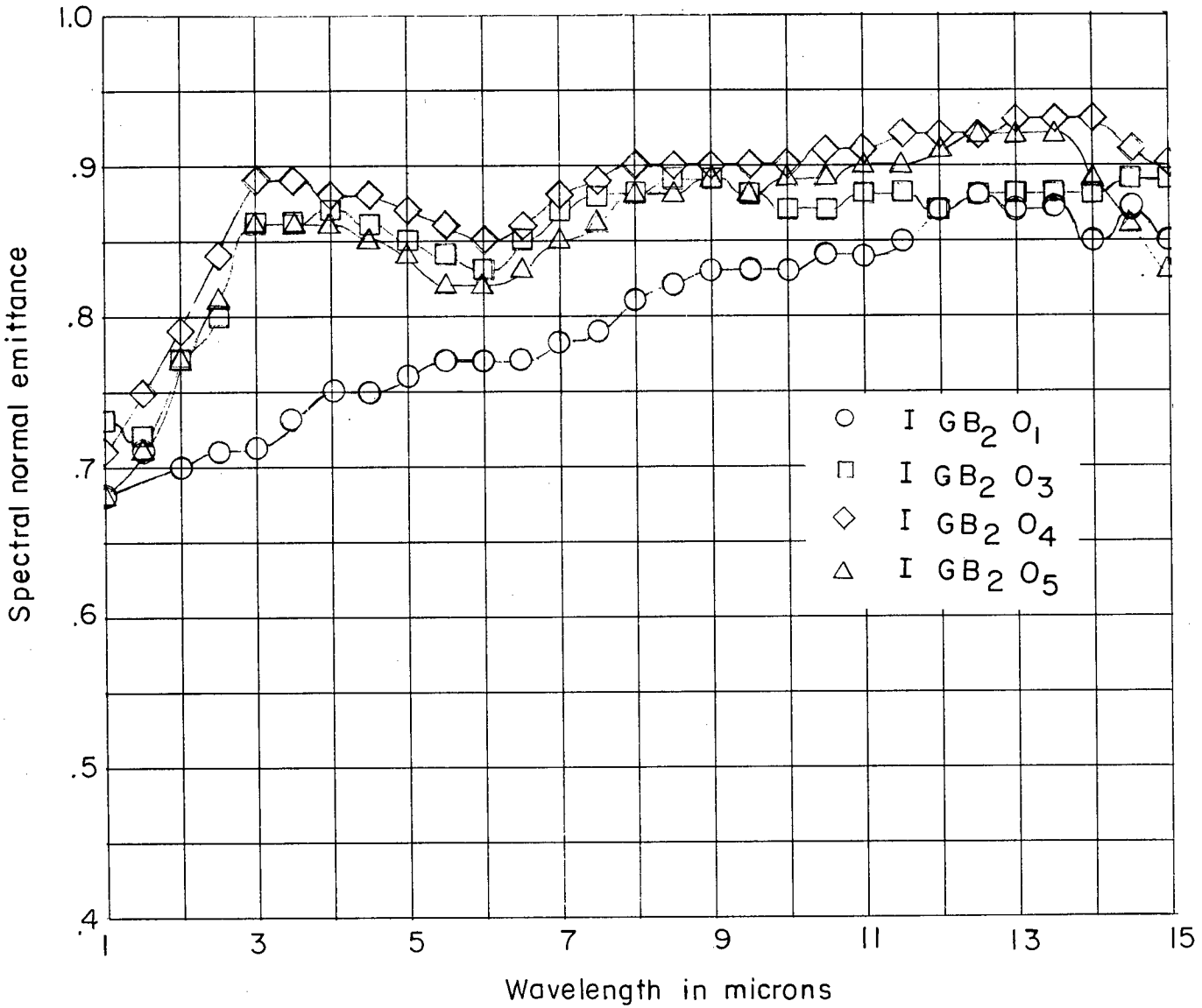

Figure 16.- Comparison of effects of oxidation time on spectral normal emittance of oxidized inconel specimens at 900° F.

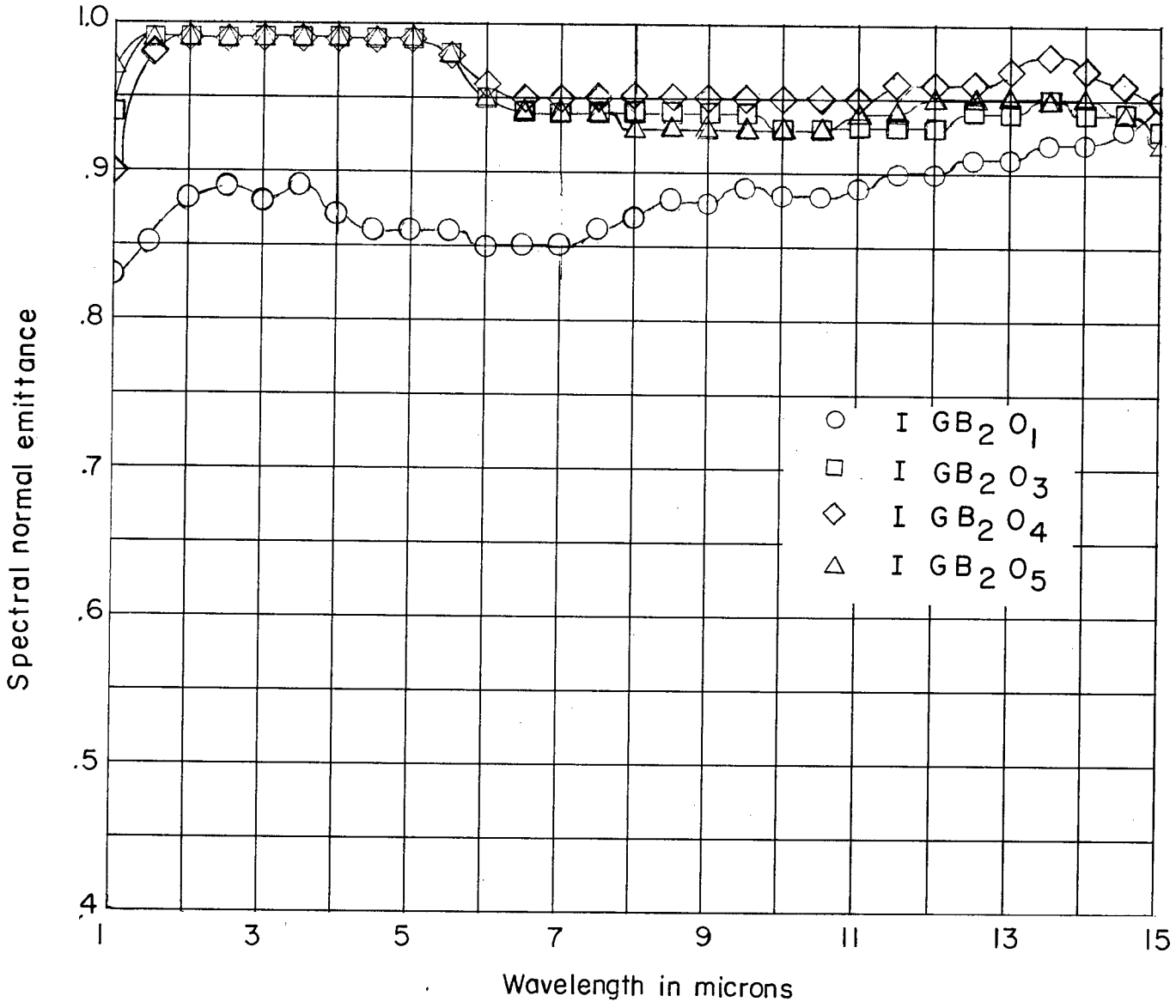

Figure 17.- Comparison of effects of oxidation time on spectral normal emittance of oxidized inconel specimens at 1,800° F.

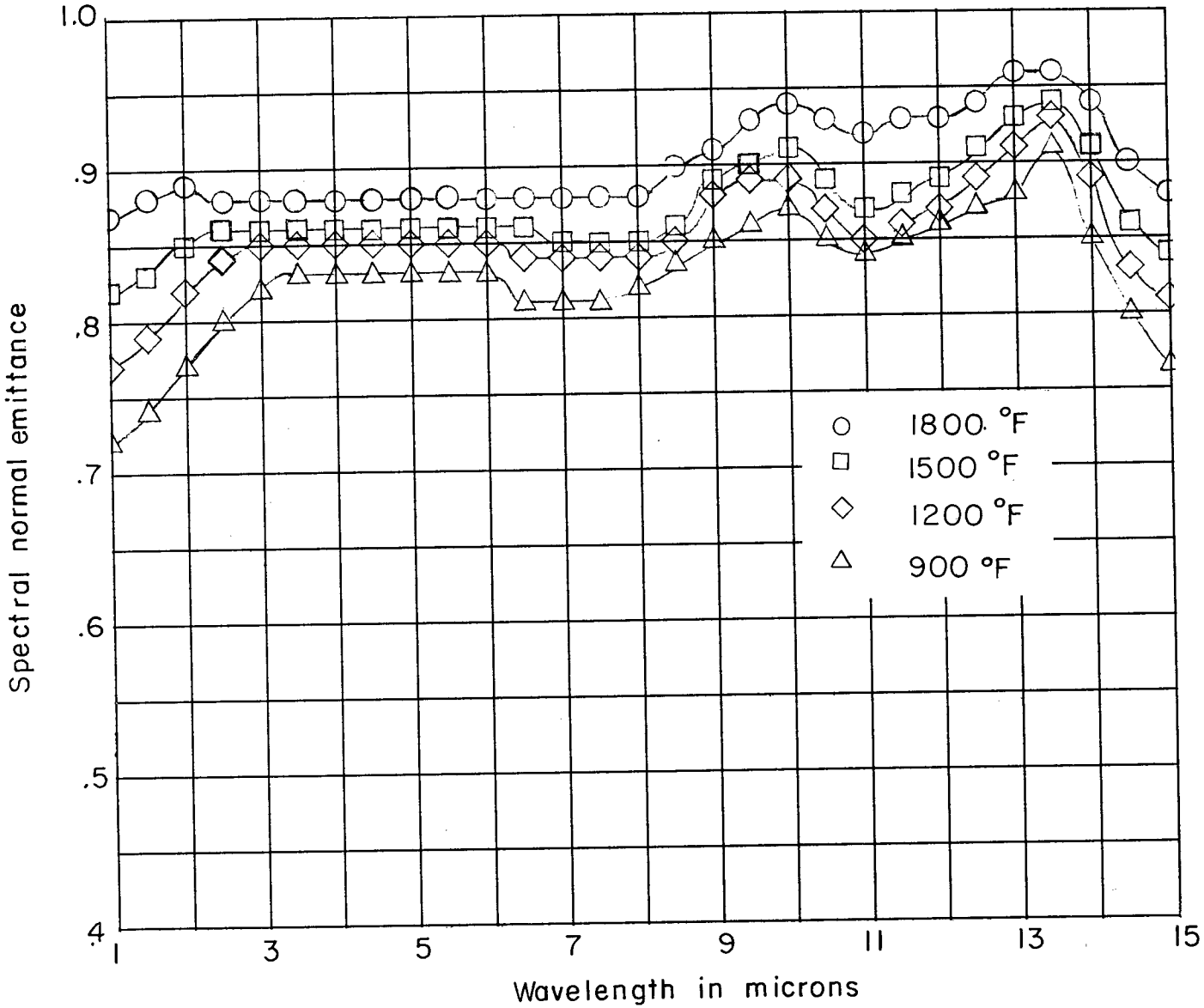

Figure 18.- Spectral normal emittance of oxidized as-received Inconel-X ($I\text{-}XARO_1$) specimens.

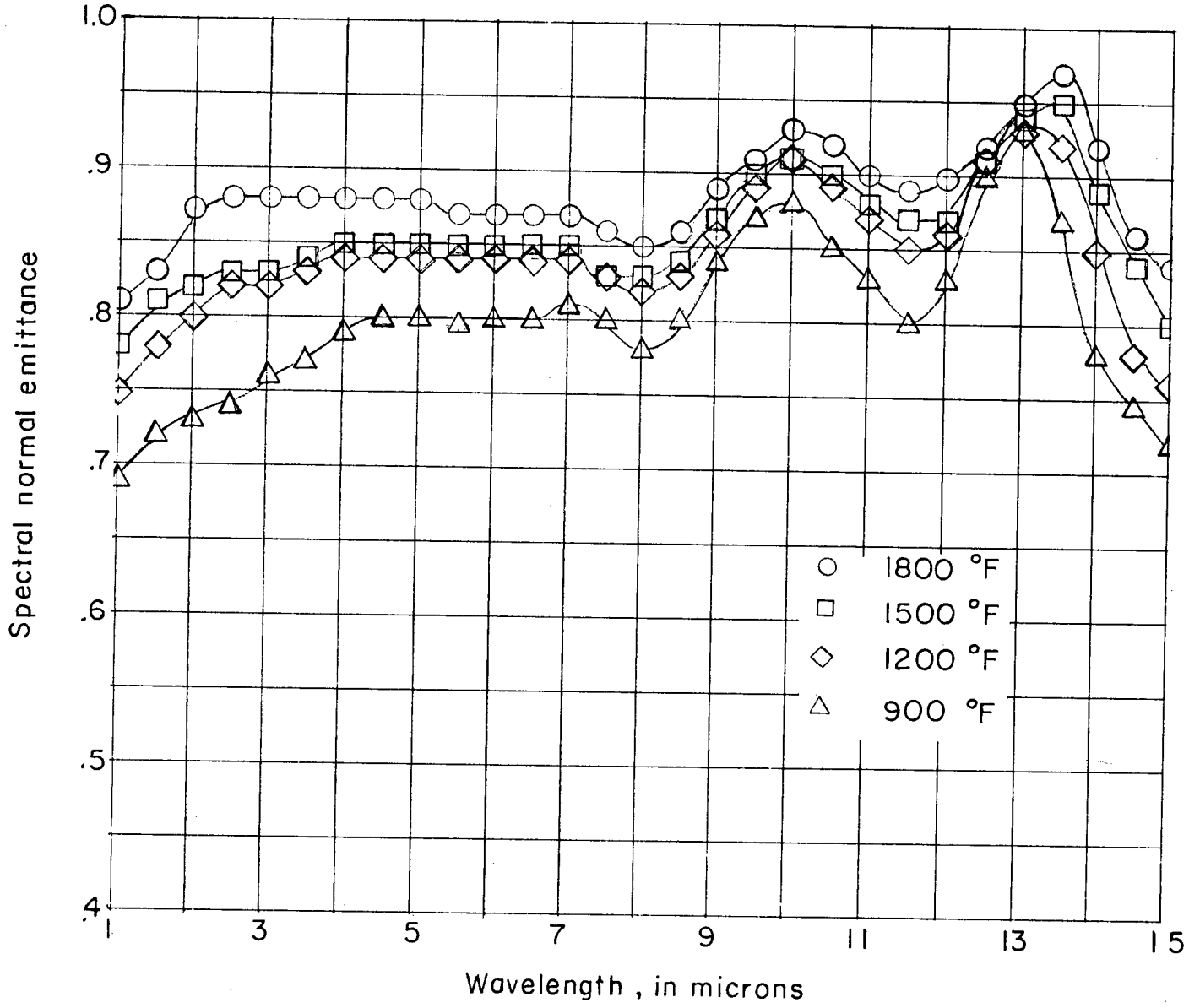

Figure 19.- Spectral normal emittance of oxidized etched Inconel-X (I-XE101) specimens.

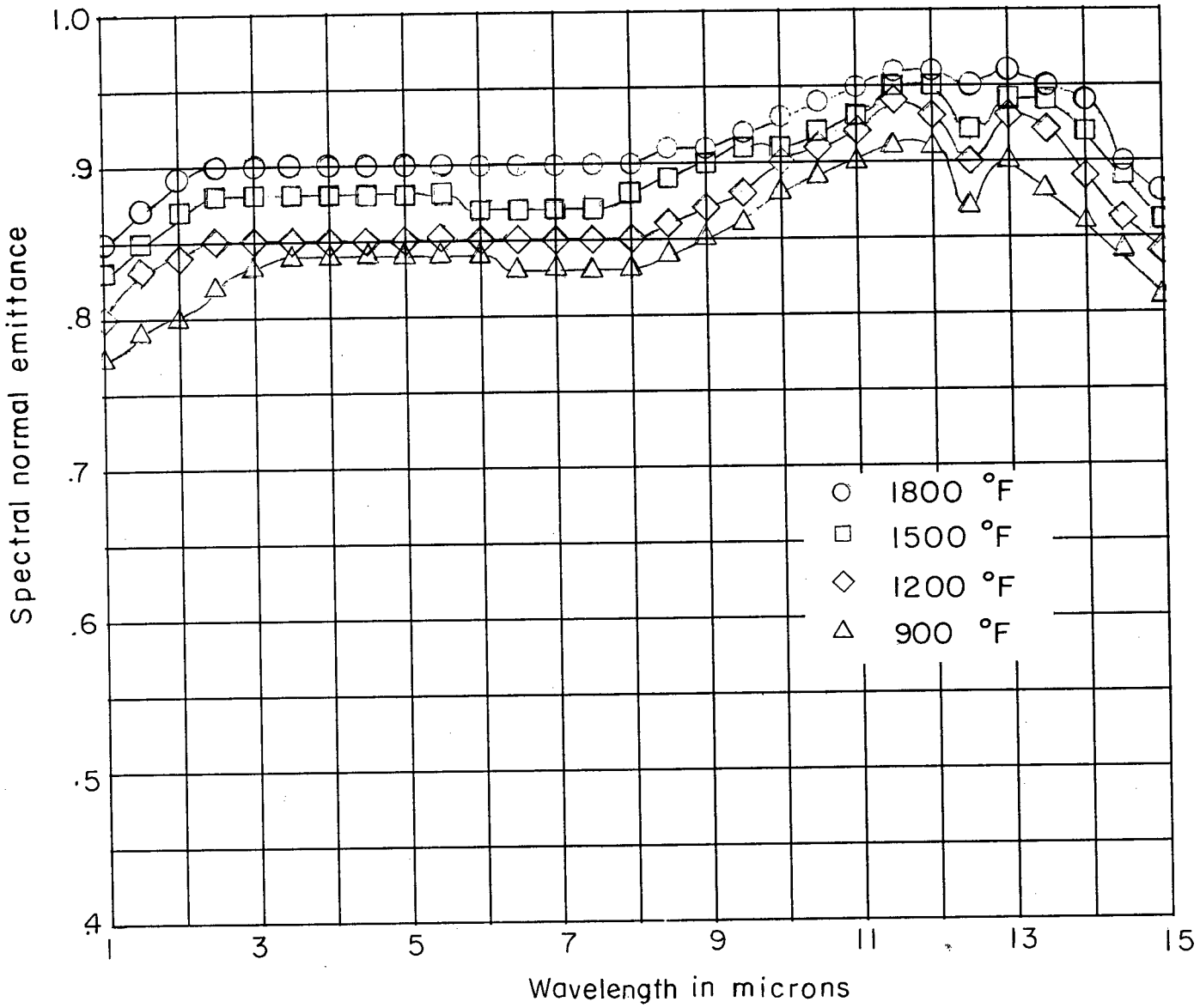

Figure 20.- Spectral normal emittance of oxidized grit-blasted Inconel-X (I-XGB$_{2}$O$_{1}$) specimens.

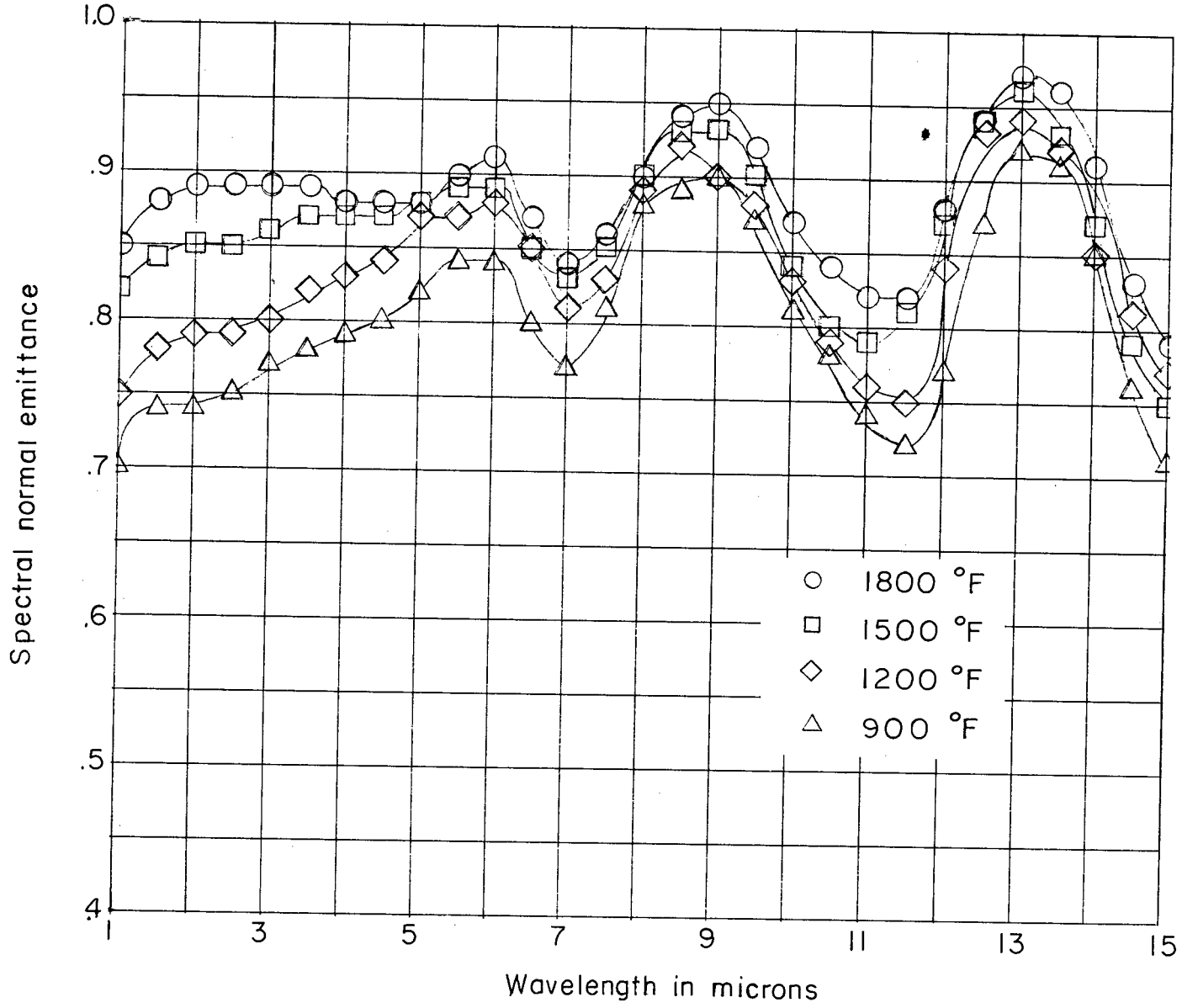

Figure 21.- Spectral normal emittance of oxidized polished Inconel-X (I-XPO$_1$) specimens.

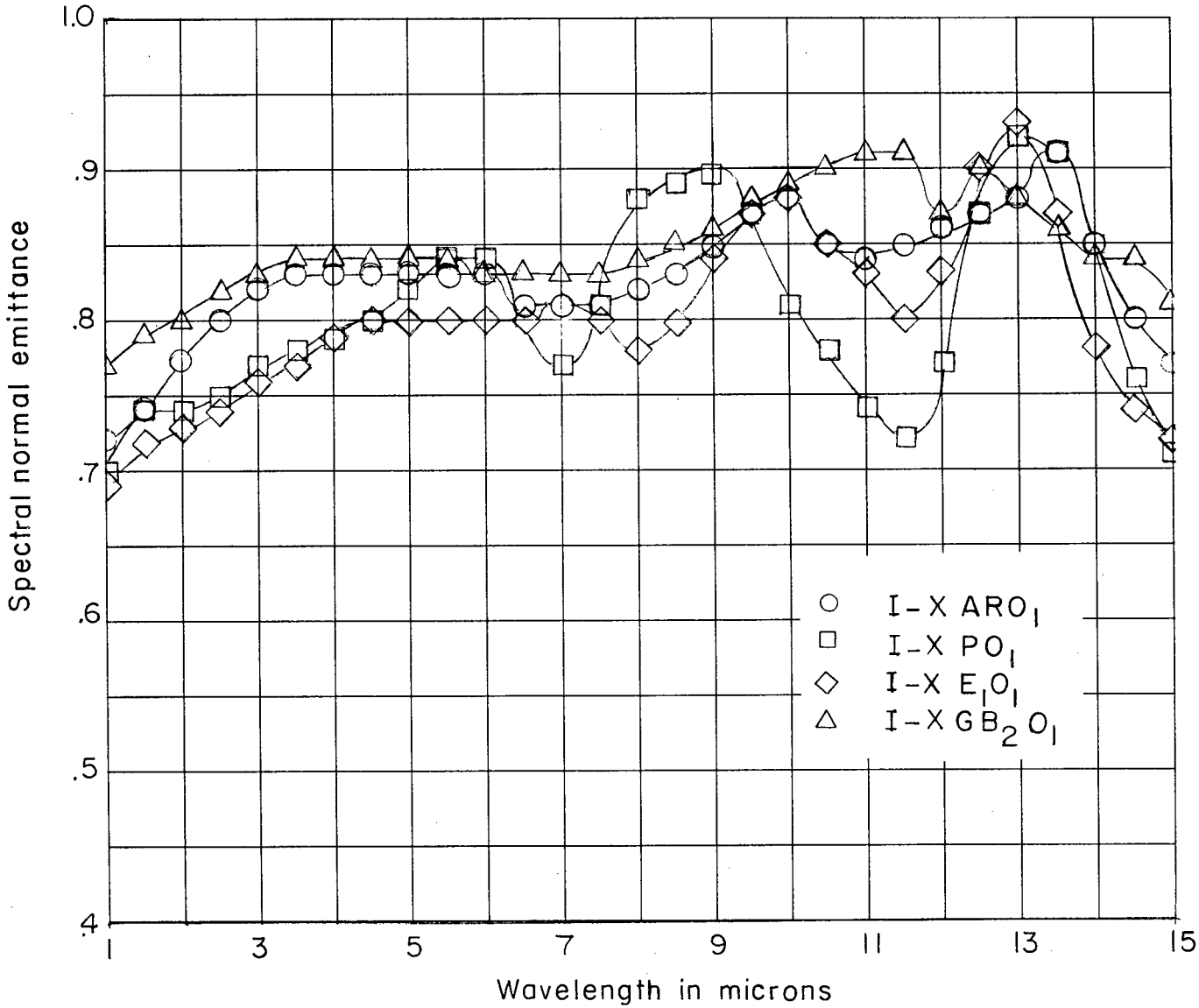

Figure 22.- Comparison of effects of preoxidation treatments on spectral normal emittance of oxidized Inconel-X specimens at 900° F.

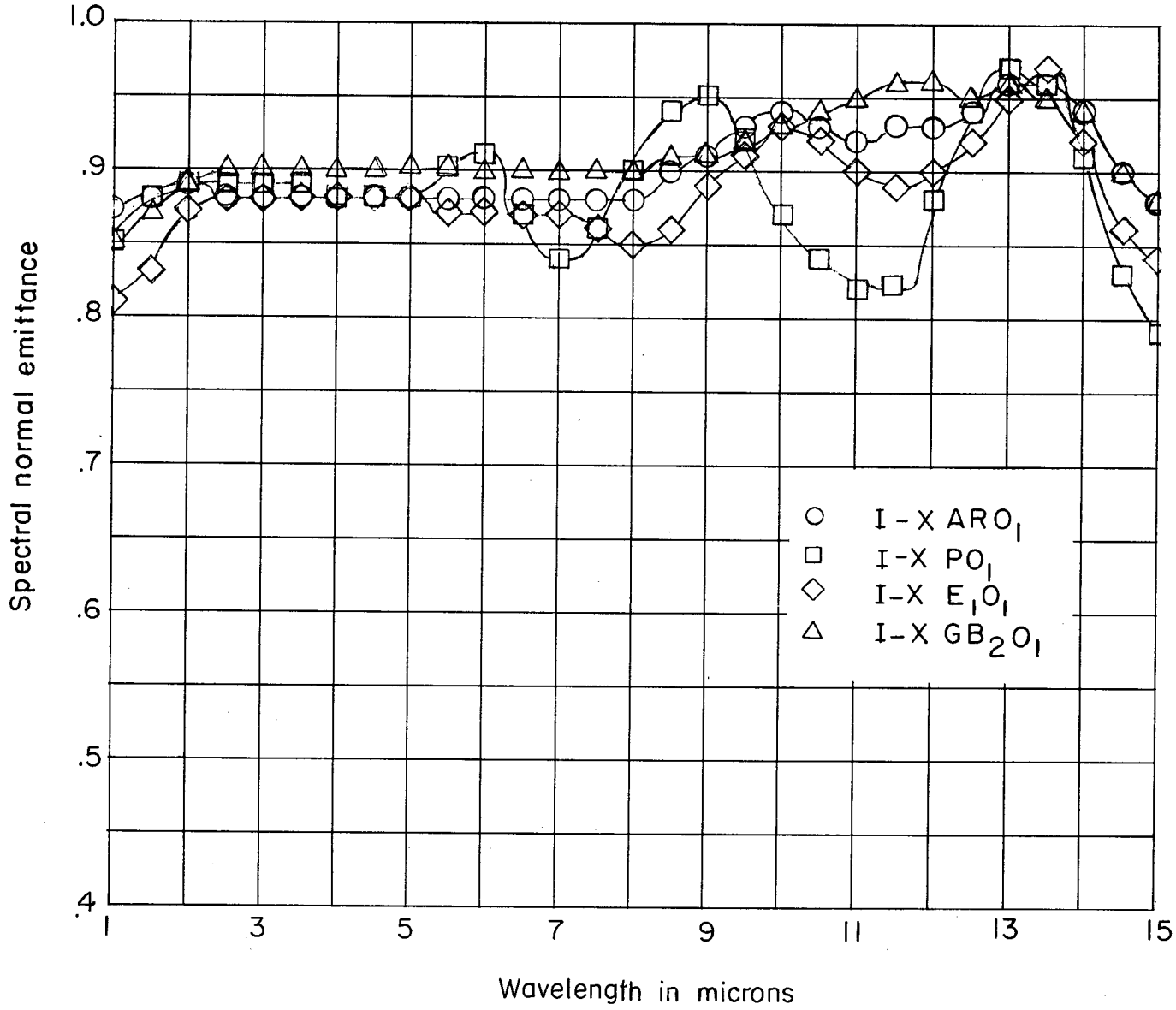

Figure 23.- Comparison of effects of preoxidation treatments on spectral normal emittance of oxidized Inconel-X specimens at 1,800° F.

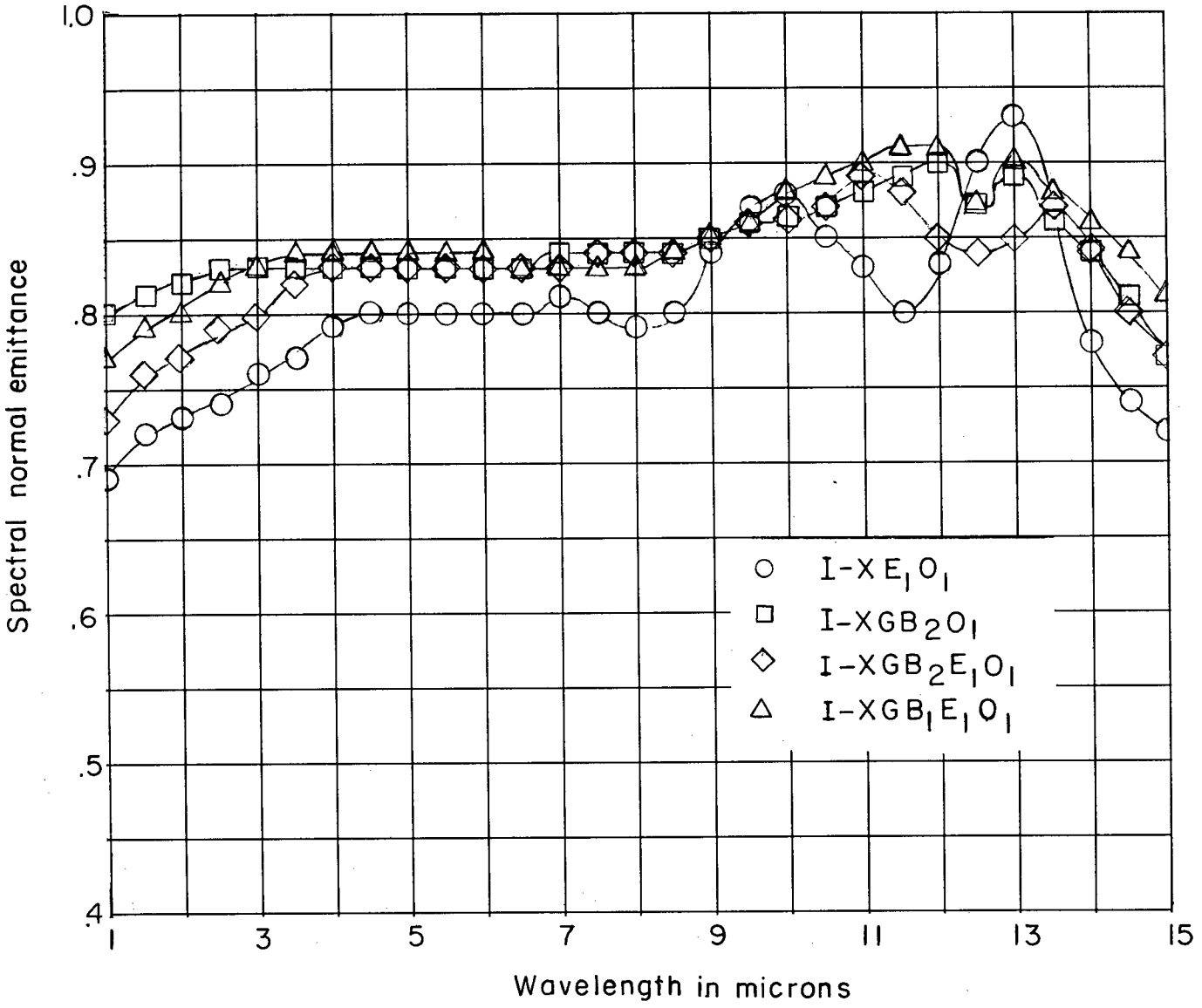

Figure 24.- Comparison of effects of combinations of preoxidation treatments on spectral normal emittance of oxidized Inconel-X specimens at 900° F.

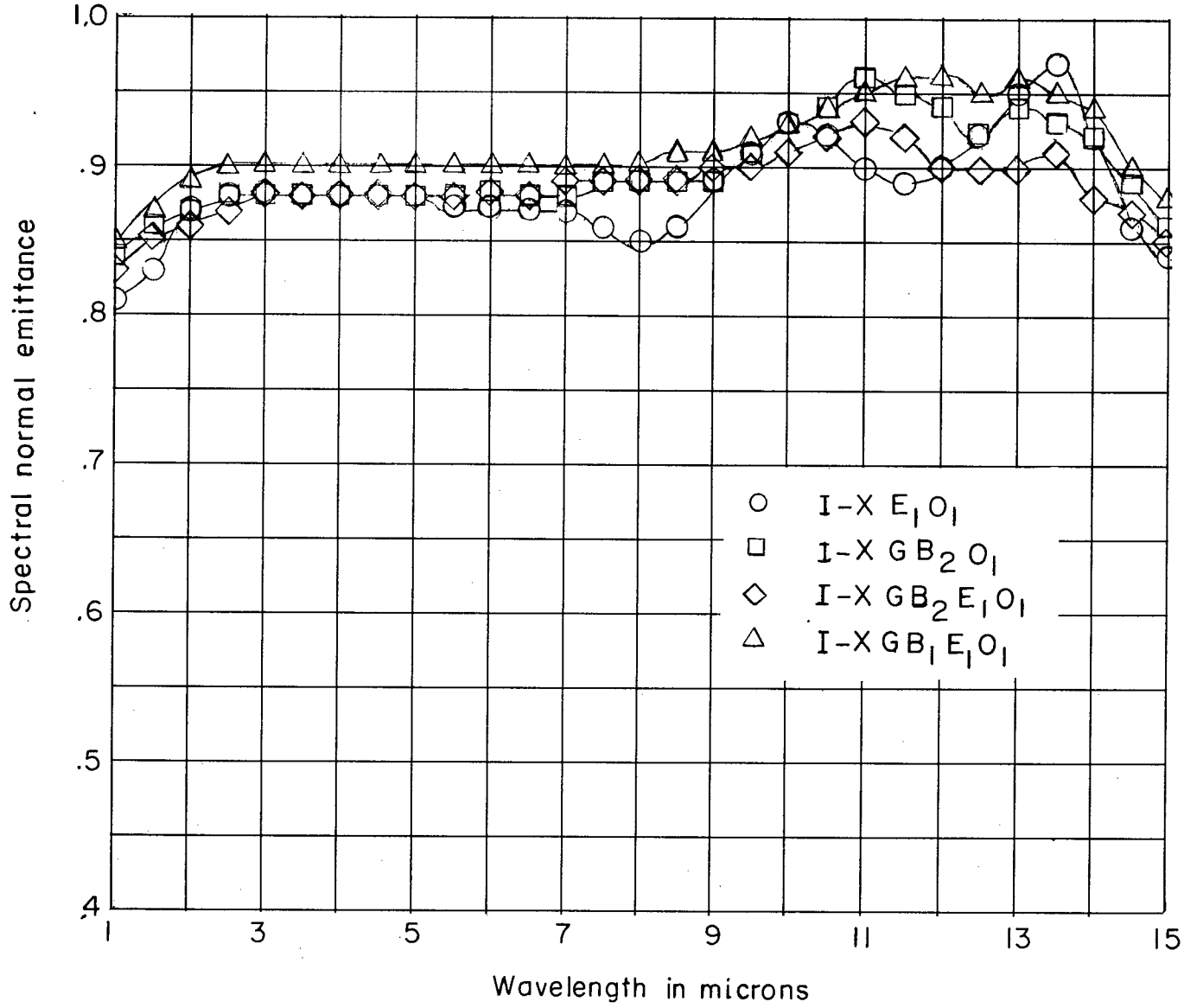

Figure 25.- Comparison of effects of combinations of preoxidation treatments on spectral normal emittance of oxidized Inconel-X specimens at 1,800° F.

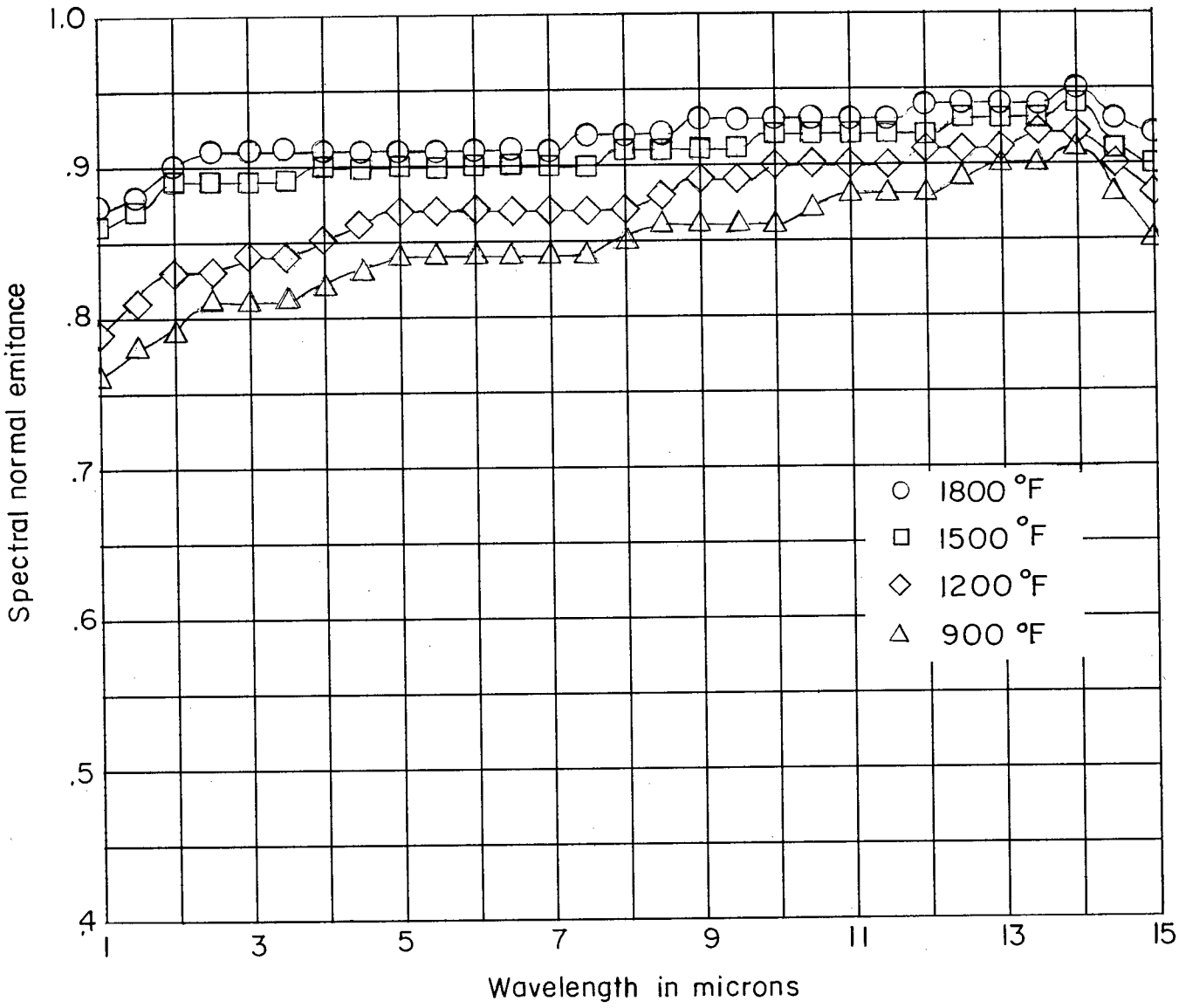

Figure 26.- Spectral normal emittance of oxidized as-received type 347 stainless-steel ($SARO_2$) specimens.

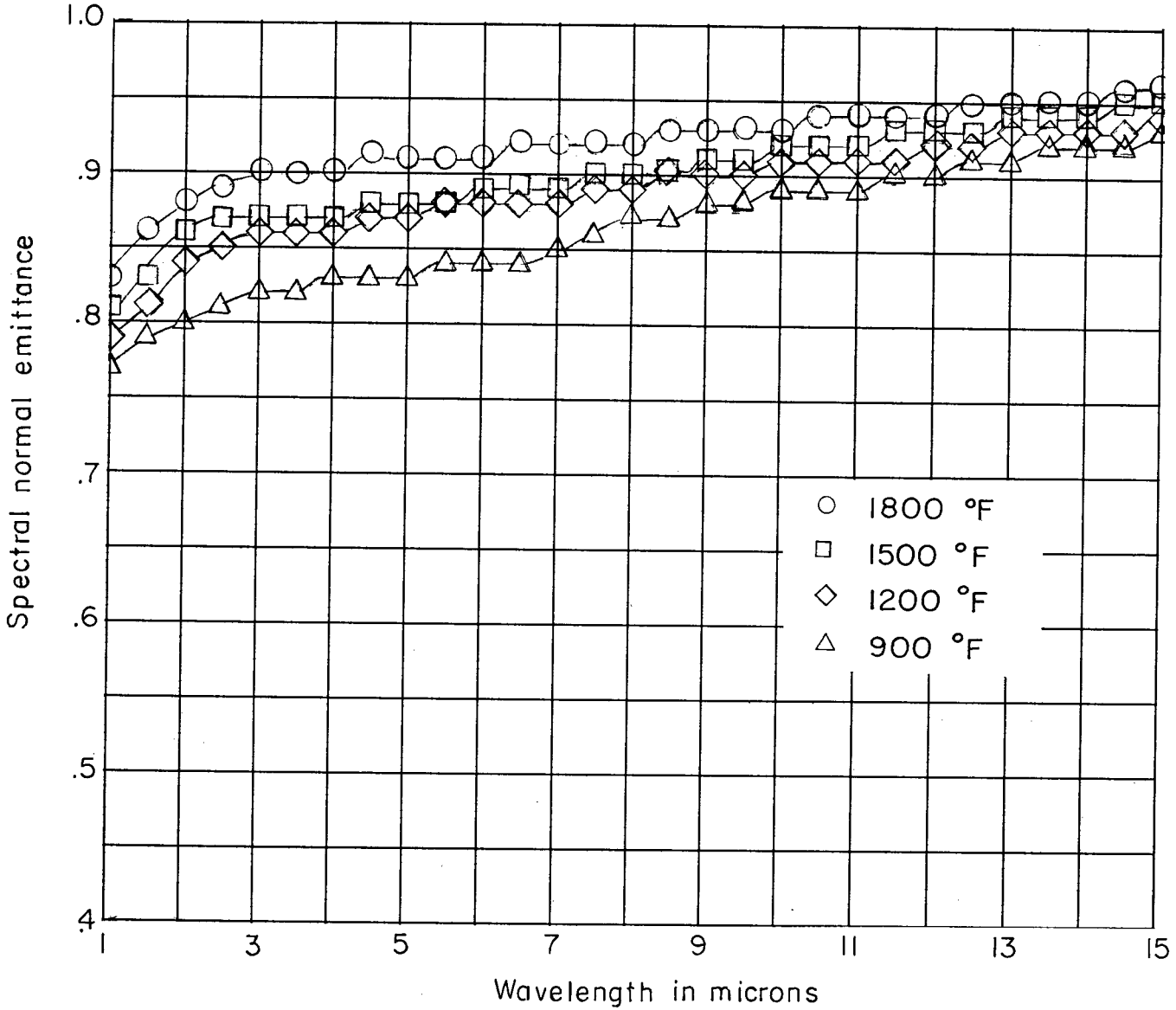

Figure 27.- Spectral normal emittance of oxidized etched type 347 stainless-steel ($SE_{2}O_{2}$) specimens.

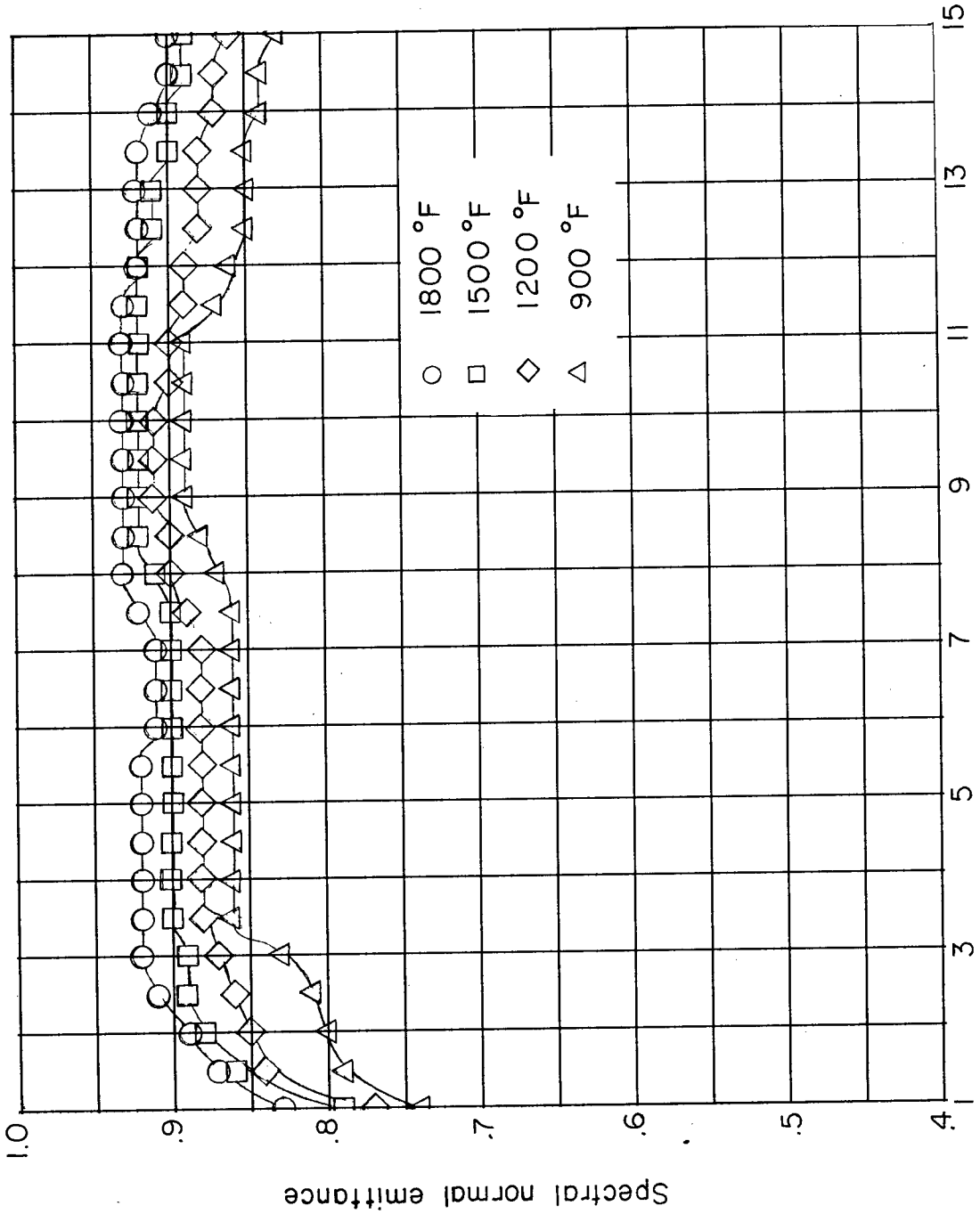

Figure 28.- Spectral normal emittance of oxidized grit-blasted type 347 stainless-steel (SGB_2O_2) specimens.

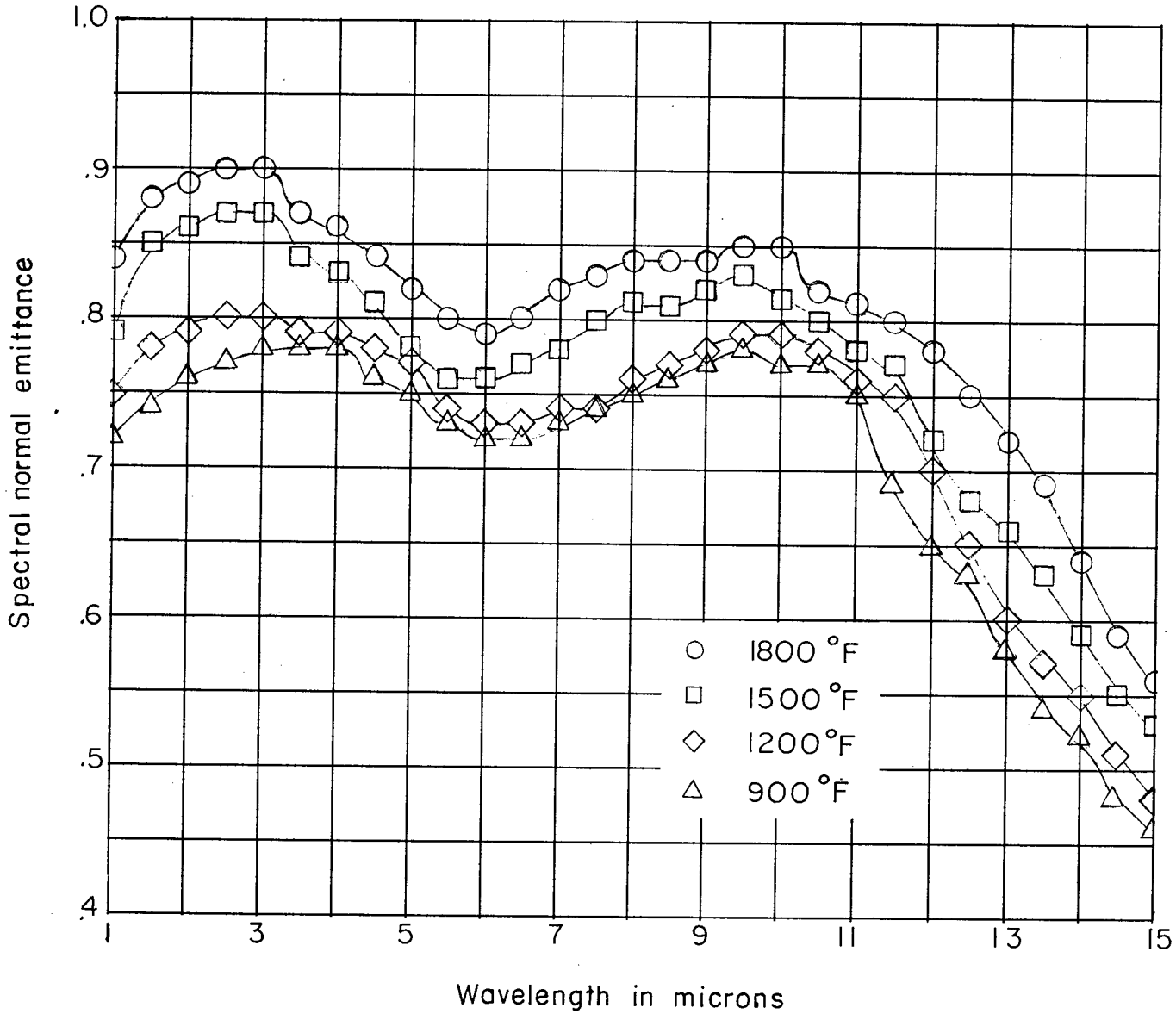

Figure 29.- Spectral normal emittance of oxidized polished type 347 stainless-steel (SPO_2) specimens.

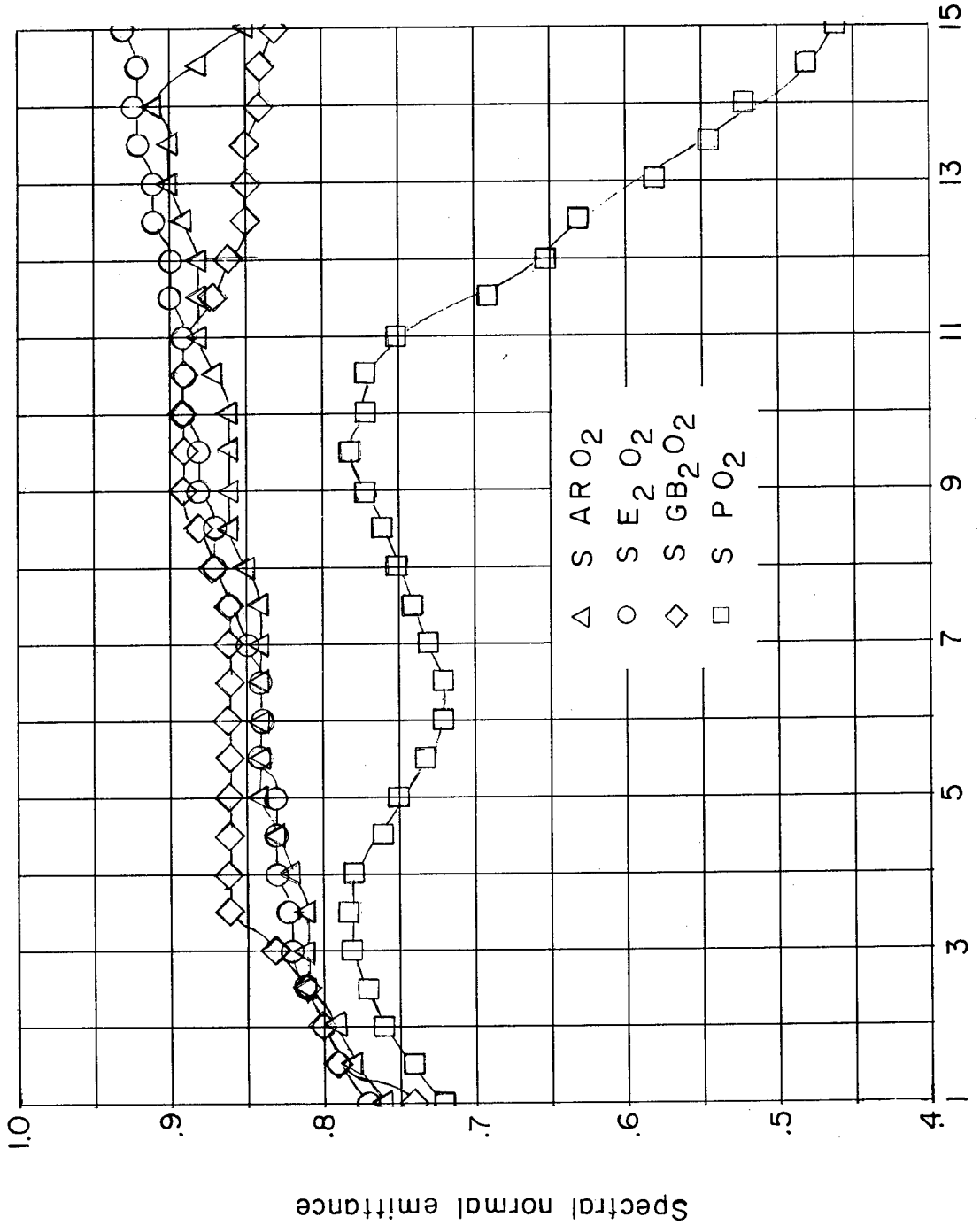

Figure 30.- Comparison of effects of preoxidation treatments on spectral normal emittance of oxidized type 347 type stainless-steel specimens at 900° F.

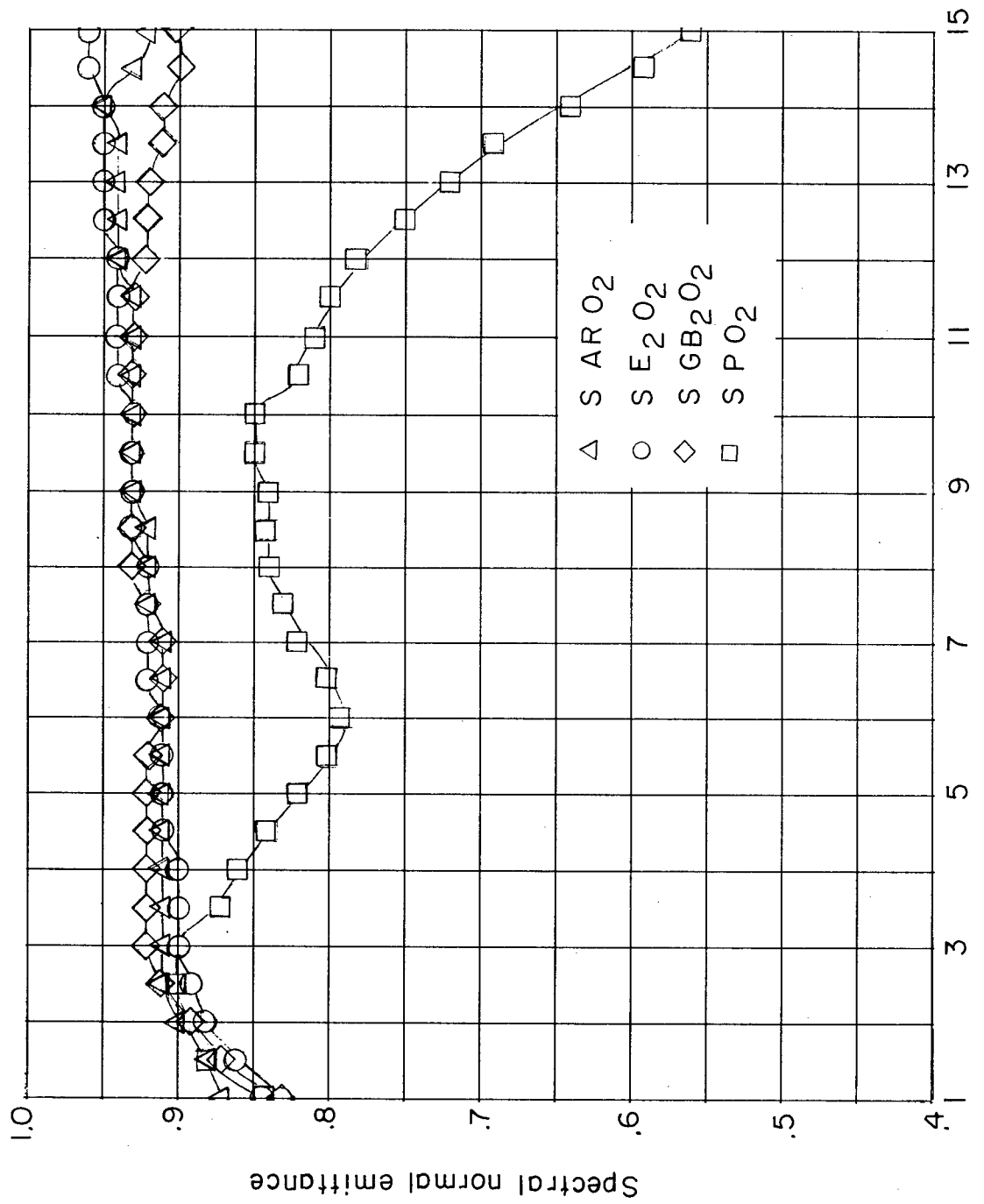

Figure 31.- Comparison of effects of preoxidation treatments on spectral normal emittance of oxidized type 347 stainless-steel specimens at 1,800° F.

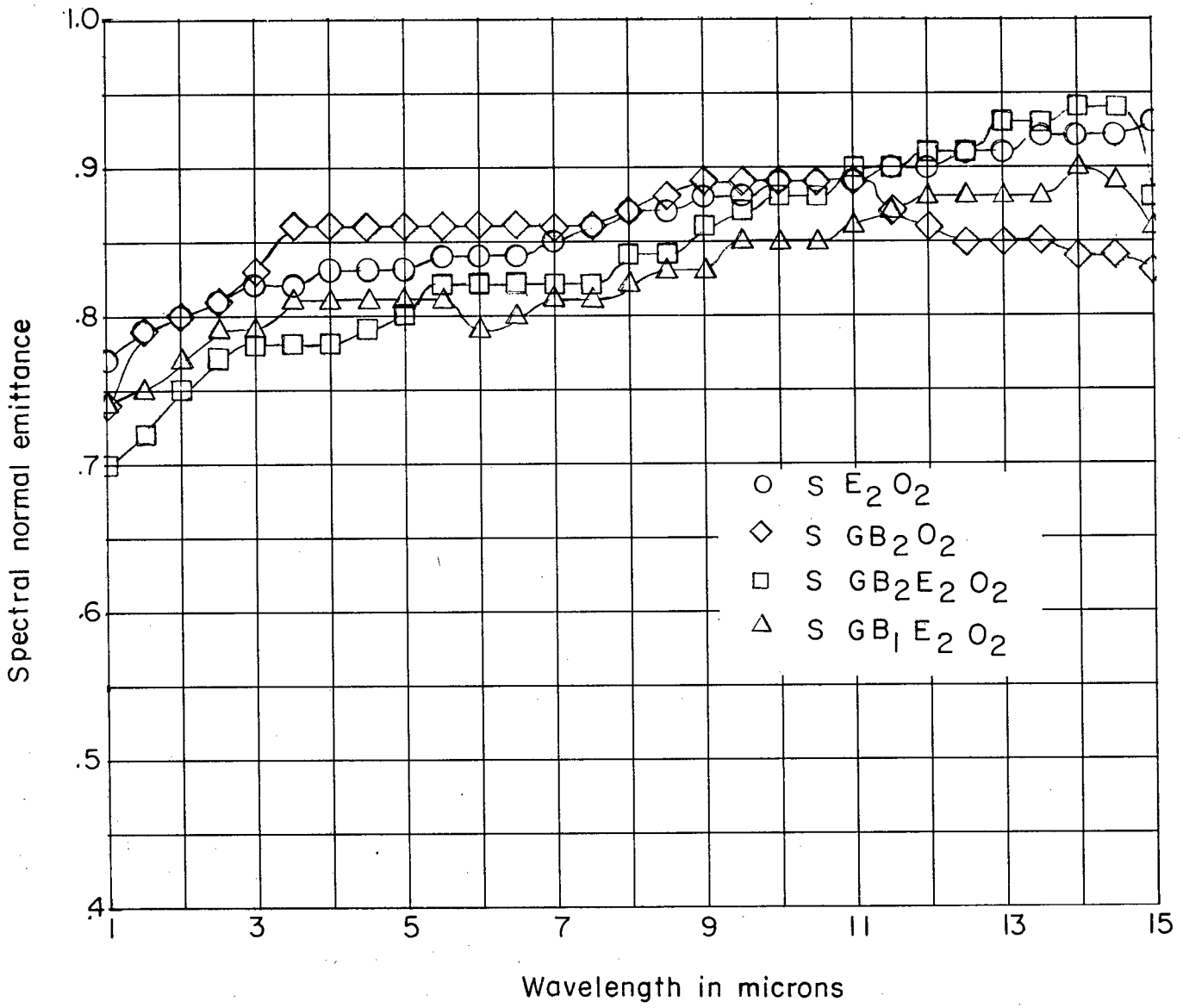

Figure 32.- Comparison of effects of combinations of preoxidation treatments on spectral normal emittance of oxidized type 347 stainless-steel specimens at 900° F.

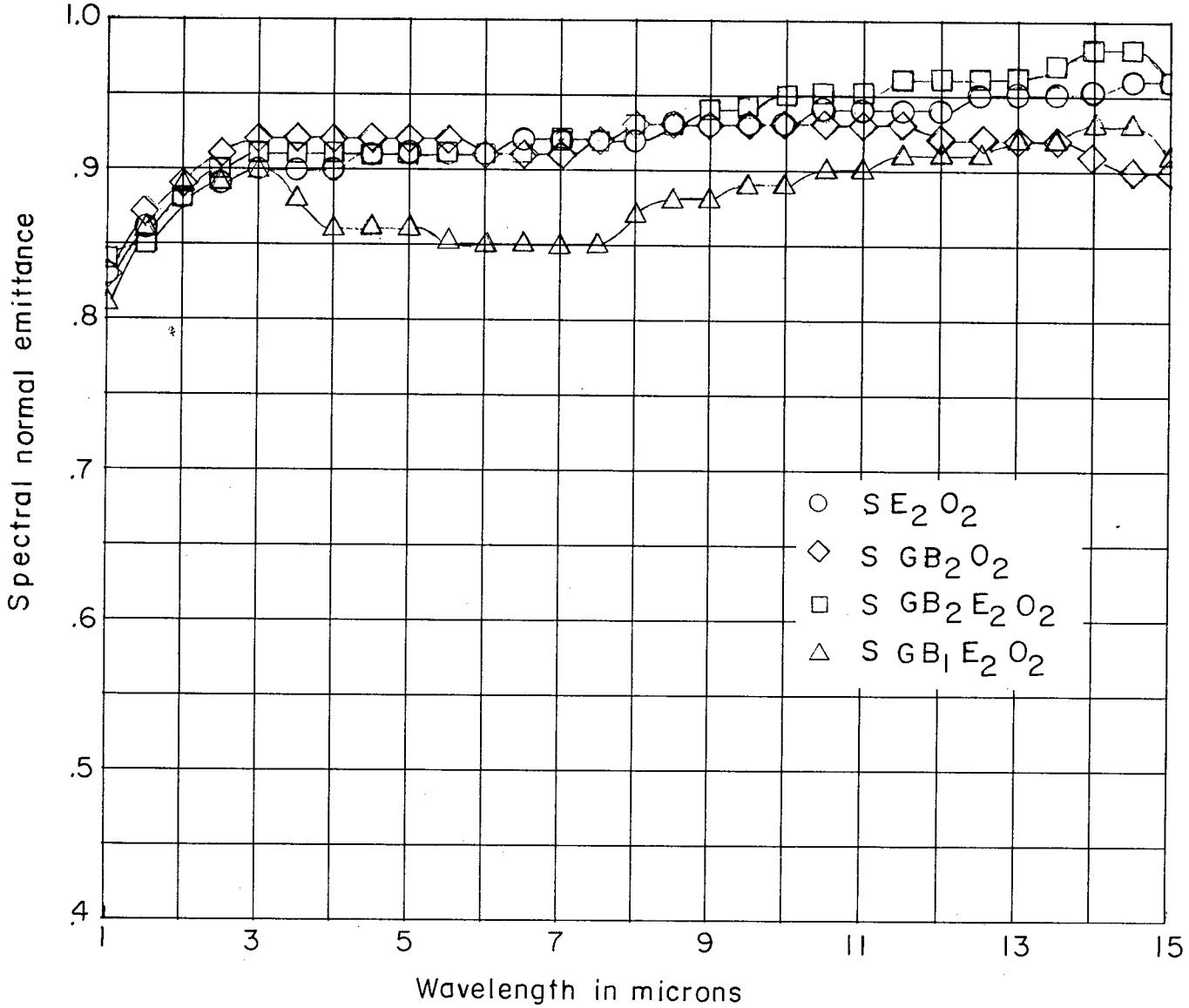

Figure 33.- Comparison of effects of combinations of preoxidation treatments on spectral normal emittance of oxidized type 347 stainless-steel specimens at 1,800° F.

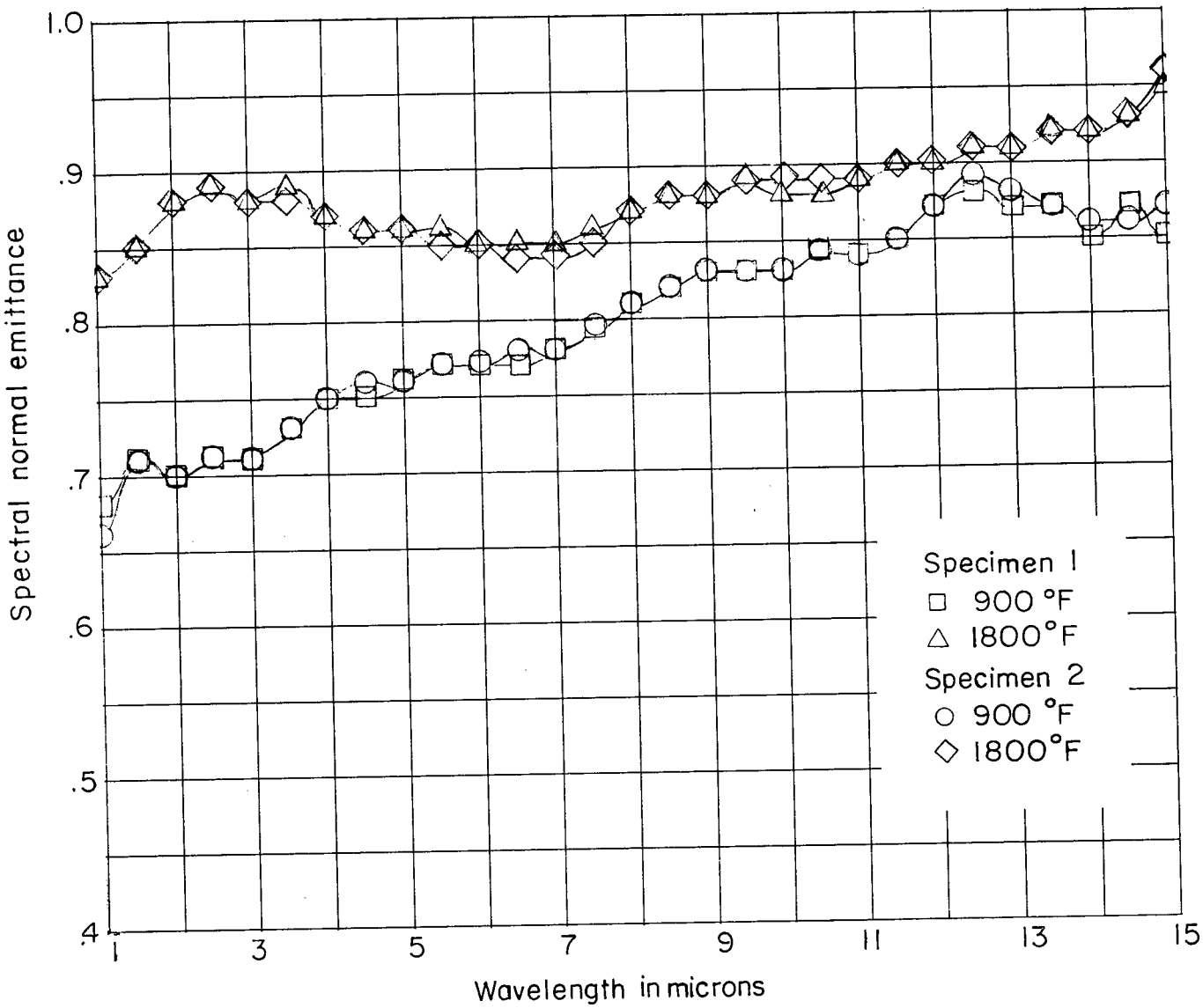

Figure 34.- Reproducibility of two oxidized grit-blasted inconel specimens (IGB_2O_1) using the same preparation and testing procedures.

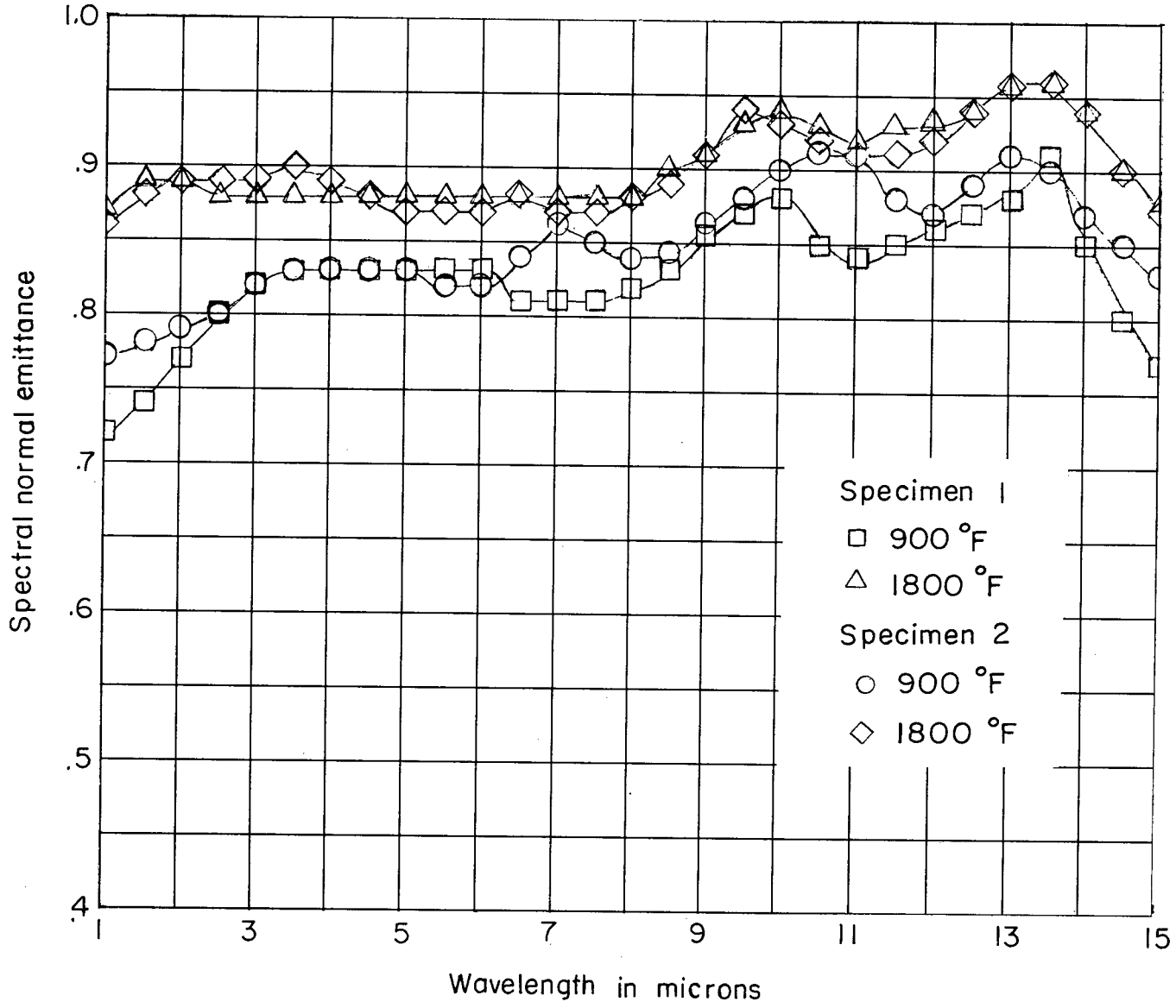

Figure 35.- Reproducibility of two oxidized as-received Inconel-X specimens (I-XARO$_1$) using the same preparation and testing procedures.

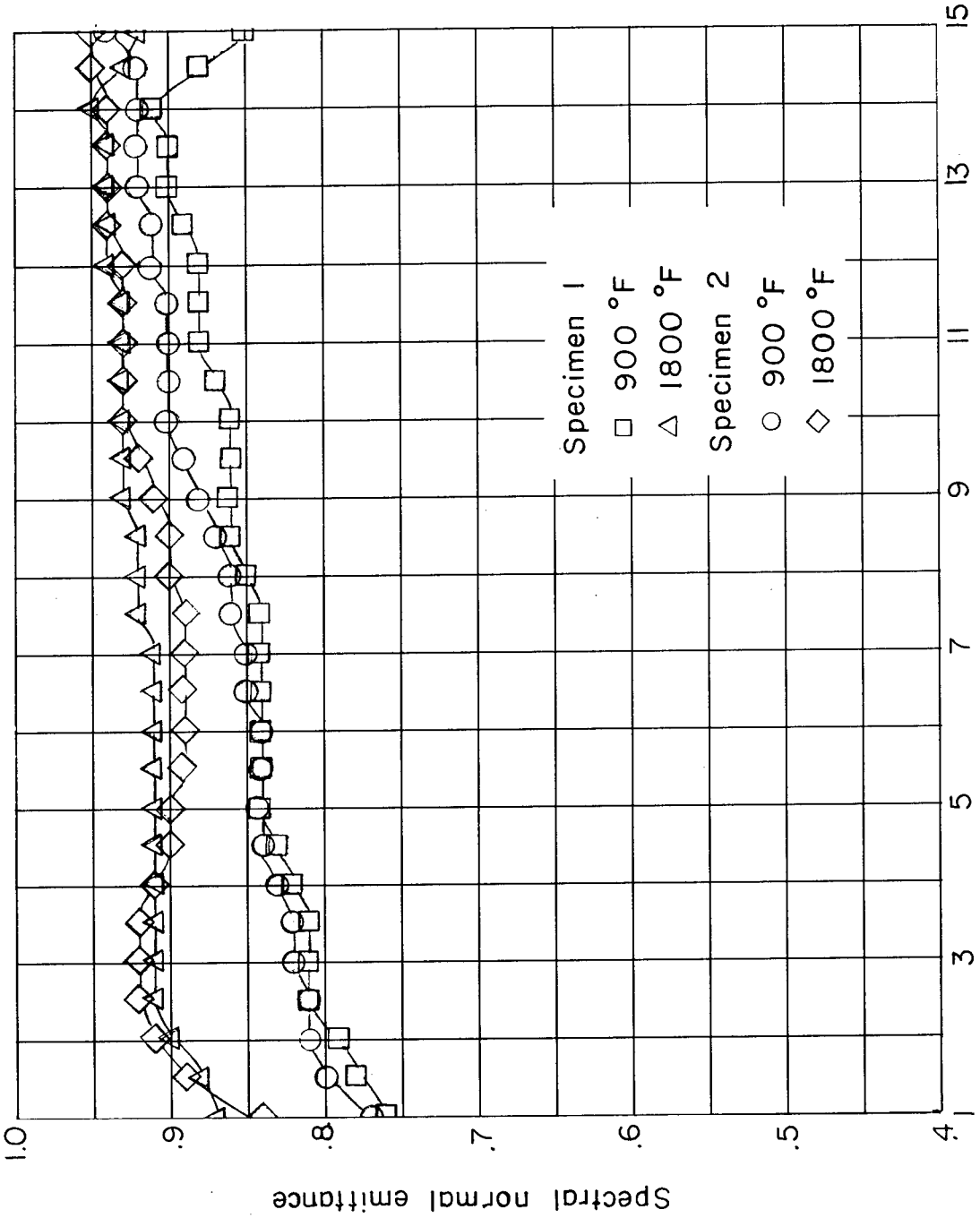

Figure 36.- Reproducibility of two oxidized etched type 347 stainless-steel specimens (SE_2O_2) using the same preparation and testing procedures.

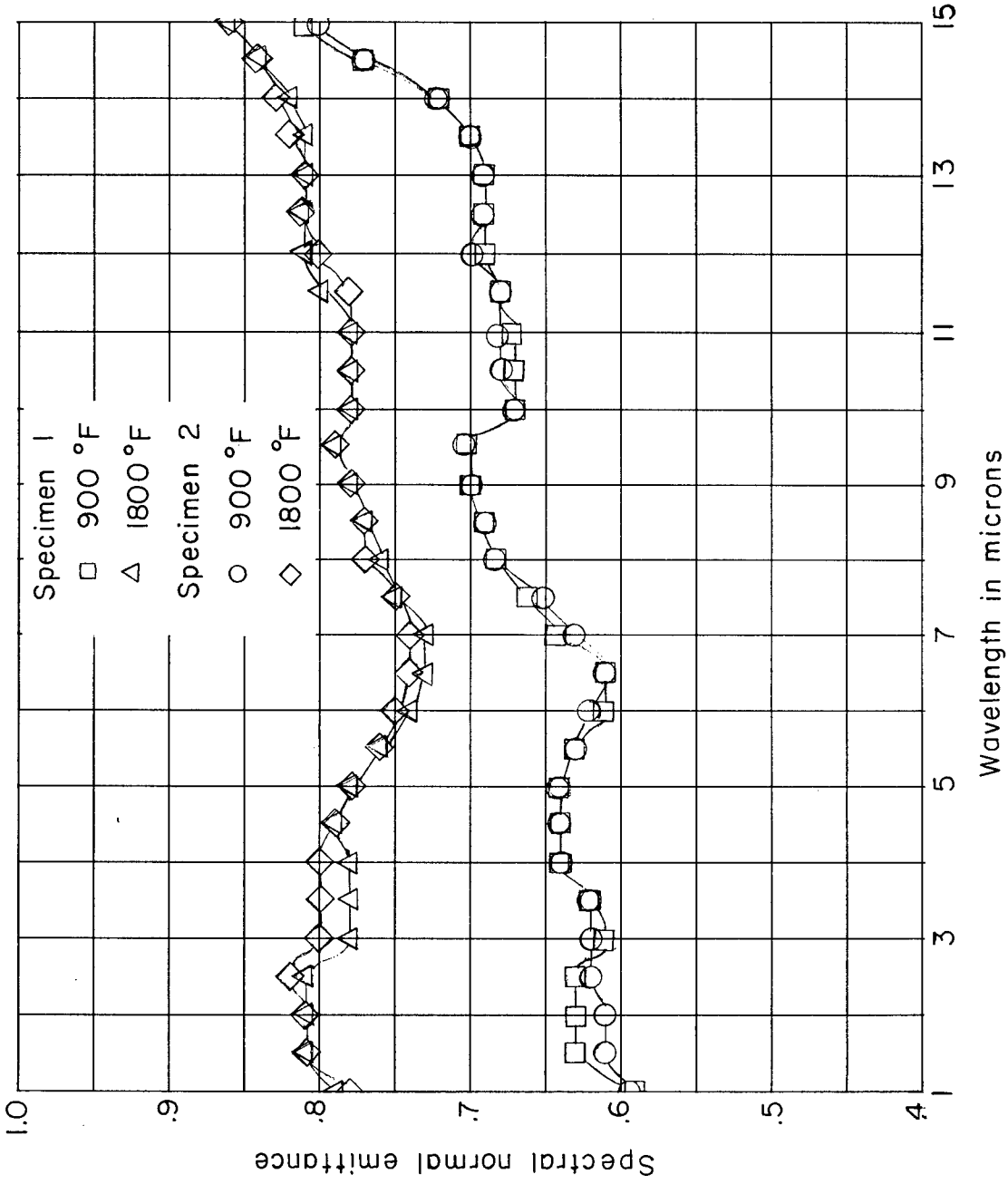

Figure 37.- Reproducibility of spectral-normal-emittance curves. Curves for two specimens representing the maximum deviation of four as-received oxidized inconel specimens ($IARO_1$) prepared and tested by the same procedure.

NASA-Langley, 1964 L-3656

NASA TN D-2300
National Aeronautics and Space Administration.
EFFECTS OF PREOXIDATION TREATMENTS ON SPECTRAL NORMAL AND TOTAL NORMAL EMITTANCE OF INCONEL, INCONEL-X, AND TYPE 347 STAINLESS STEEL. Wayne S. Slemp. July 1964. 56p. OTS price, $1.50.
(NASA TECHNICAL NOTE D-2300)

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