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Effect of electropolishing on vacuum furnace design

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Abstract

The use of thermal shields of materials having low emissivity in vacuum furnaces is well-known. However, the surface condition of the heat shields is one of the most important factors governing their efficiency as radiation resistances. The emissivity of the thermal shields dictates the power rating of the heaters in furnace design. The unpolished materials used in the heater tests showed poor performance leading to loss of a significant percentage of the input power. The present work deals with the refurbishment of the radiation heat shields used in a furnace for heating graphite structure. The effect of refurbishment of the heat shields by the buffing and subsequently electropolishing was found to improve the performance of the shields as heat reflectors. The composition of the electrolyte was chosen in such a way that the large shields of Mo, Inconel and SS can be polished using the same reagents in different ratios. The present work deals with the development of a standard electropolishing procedure for large metallic sheets and subsequently qualifying them by roughness and emissivity measurements. The improvement noted in the shielding efficiency of the furnace in the subsequent runs is also discussed here.

Keywords

Thermal shields; Etching; Emissivity

Introduction

The dominant mode of heat transfer in vacuum furnaces is radiation. The use of radiation heat shields to reduce the power loss from the hearth of a furnace is well known [1-4]. A set of heat shields made of various materials and assembled with minimum inter-shield separation, increases

the net thermal resistance of the set. The most important factor governing the performance of each heat shield in the pack is the emissivity of both the surfaces, which, in turn, depends on their surface condition [1].

The vacuum furnace used in this study, has a pack of nine heat-shields as shown in figure 1. The pack of shields is designed to: (a) support heaters and (b) enclose the graphite hearth except a few view-ports. The innermost shield facing the hearth is of molybdenum and the next three shields were inconel-600 and the rest SS-304L. The Mo heater wires are laid on the innermost shield facing the graphite structure. The power input to the heaters is used to maintain the graphite structure at a temperature of 1500 K. The heat shields help in maintaining the hot zone at 1500 K without increasing the temperature of the chamber wall beyond 400 K. The vacuum chamber is maintained at a vacuum of the order of 10^{-5} mbar.

The primary objective of the experiment was to maintain the graphite hearth at a temperature of 1500 K. However, experiments with the unpolished heat shields as received from the fabricator failed to achieve the target temperature. Heat was being lost from the furnace primarily due to the high emissivity of the shields. The graphite structure could reach a temperature of 1200 K at 124 kW. On extrapolation, the required power to achieve 1500K was 320kW.

On further analysis, the presence of MoO and MoC was detected on the surface of the innermost shield. Carbon was present on all surfaces of the heat shields. The oxidation and the carburization is a result of the outgassed species from the graphite hearth. The data from the residual gas analyser (RGA) showed the presence of methane and carbon in significant quantities during the experiment. The presence of carbon and the formation of carbide resulted in an increase in their emissivity and therefore, loss of input power. The unpolished surface provides a larger area for carbon deposition and carbide formation and hence acts as a catalyst in the deterioration of its emissivity.

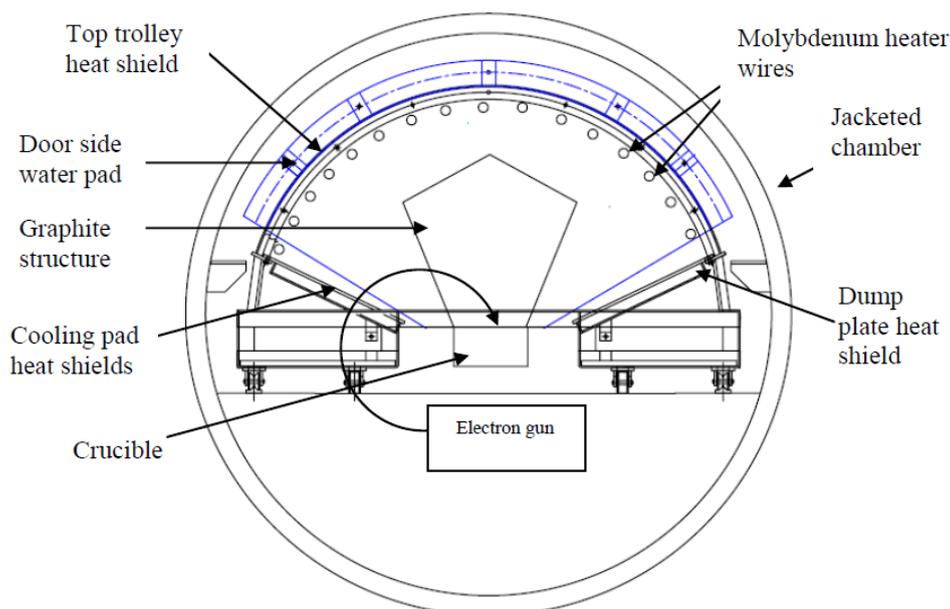


Figure 1. Schematic diagram of the furnace

The effective emissivity or reflectivity is a strong function of the surface finish [12-15]. As the temperature of the shield increases, the surface roughness needs to be significantly less than the peak wavelength of the heat radiation at working temperature to obtain high values of reflectivity. This calls for a highly polished surface.

Electropolishing is essentially the process of anodically smoothing of a surface in a suitable electrolyte. It removes the microscopic roughness on the surface, eliminating imperfections which trap and contain contaminants, in a more controlled manner compared to the other forms of chemical cleaning. Hence, electropolishing was chosen as the method of refurbishment of these shields. In short, the object to be electropolished is immersed in an electrolyte of an optimized composition and subjected to direct current [8].

Experimental

A flowchart summarizing the experimental procedure is given in Figure 2. It may be noted here that the range of average roughness values that electropolishing can achieve is 0.8 to 0.1 microns [18]. Therefore, 0.2 microns was selected for the roughness check.

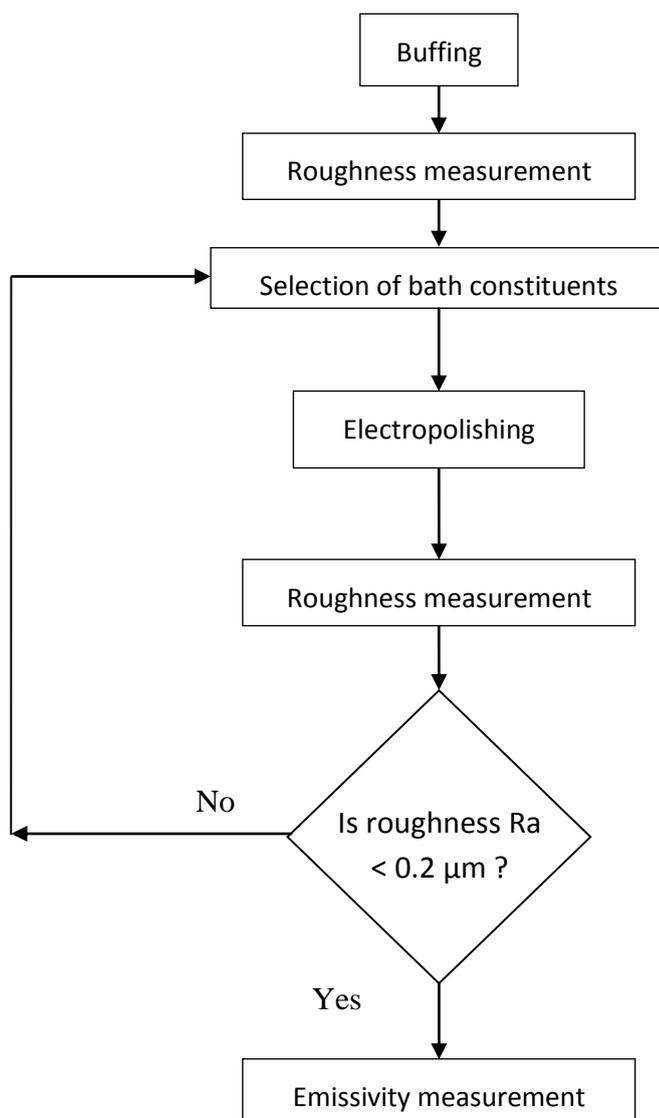


Figure 2: Flowchart of the experimental procedure

Selection of the bath constituents

There is a wide spectrum of electrolytes used in industrial application for electropolishing. The stainless steel has established electropolishing procedures with sulfuric acid and phosphoric acid [17]. However, some of the electrolytes reported for molybdenum and inconel (like potassium dichromate, etc) are too hazardous to be handled on a large scale [6-7]. Moreover, it is convenient

to use the same bath constituents for polishing all the radiation heat shields by changing the ratio of the acids, instead of using separate baths and chemicals for separate materials. Since the process was to be carried out on large shields of 2000×500 mm area, a few trials were carried out on small samples to determine the optimum electropolishing conditions using the industrial solvents like sulfuric acid and phosphoric acid.

Pretreatment

Poor base metal conditions can result in less than optimum electropolished finishes. These flaws are revealed by electropolishing. Hence suitable pretreatment is required. In view of this, the shields were buffed with buffing wheel of grades 60 and 120. After the mechanical polishing and removal of burrs and sharp edges, the samples were washed in running tap water. The micrographs of the samples were obtained. Its roughness was measured and recorded.

Electropolishing

An electrolytic bath of dimensions 1500×1500×1000 mm was filled with a given composition of electrolyte. The copper plates of dimensions 400×200×2 mm was used as cathode. The voltage was set at a given value and the surface of the specimen (200×80×1 mm) was inspected after an interval of 3 minutes using a magnifying glass. The distance between anode and cathode was 800 mm. The current, voltage and time were noted. Temperature of the bath was monitored and found to rise to a steady state maximum temperature of 45 °C. It may be noted that there was no external agitation in the bath. After the specimen was removed from the bath, it was put under running tap water and dried in air for half an hour.

Emissivity measurement

One of the objectives of heat shield cleaning is to reduce the emissivity so that the heat loss due to absorption in the heat shields is reduced. The samples of Mo, Inconel and SS 304 sheets used in the heater runs and those obtained after electropolishing were made into filaments of suitable size. The bare wires of chromel and alumel are spot-welded on the filament and used as a thermocouple for measuring temperature as shown in Figure 3. The transmissivity of the glass window is known. Hence the difference in the temperature readings of the thermocouple (T_{actual}) was compared to the temperature measured by the pyrometer (T_{measured}) and the emissivity value ϵ of the filament was determined at 0.65 μm wavelength. The following expression was used for estimating emissivity [4]:

$$\frac{1}{T_{\text{measured}}} - \frac{1}{T_{\text{actual}}} = \frac{\lambda \ln(\tau\epsilon)}{1.4388}$$



Figure 3. (a) Filaments made out of the heat shields (b) Hot filament as viewed by the pyrometer

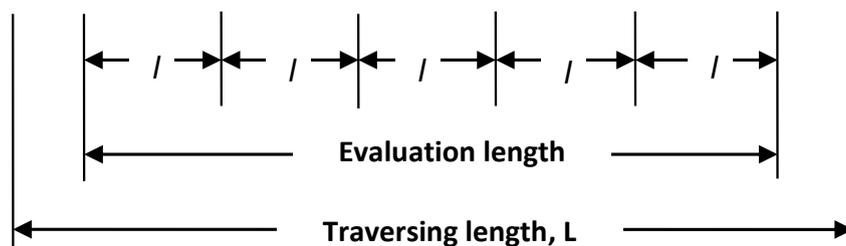


Figure 4. Typical surface profile and evaluation length

Roughness measurement

A token of area 50×50mm was made from each material and roughness was measured before and after electropolishing. The specimen is placed on a paper placed on a vibration less table. One edge of the specimen is aligned to the stand of a calibrated roughness meter and the stylus was used to scan the surface over a tracing length of 17.5 mm. The tracing length consists of pre-travel L_v , post travel L_n , and the evaluation length L_m . The evaluation length (in this case, 12.5 mm) consists of n ($n = 5$) number of sampling lengths (2.5 mm). The surface profile was recorded and analysed. A typical surface profile is shown in Figure 4.

Results and Discussion

Condition before electropolishing

It is observed that the surface condition of the virgin heat shields brought from the mill were not good enough to offer low value of emissivity. The imperfections acted as contamination traps and led to the deterioration of emissivity as the power loss from the hot zone was increased. The micrographs of the unpolished heat shields are shown in Figure 5.

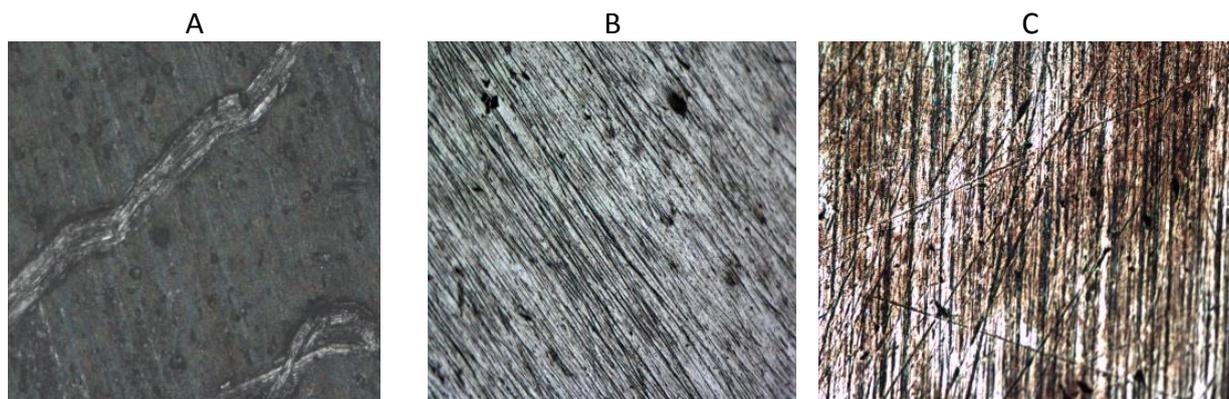


Figure 5. Micrographs of the virgin heat-shields: A) Stainless steel B) Inconel C) Mo

Electropolishing

The optimum electrolytic conditions for molybdenum, inconel and steel are given in Table 1. Several trials were carried out to determine the optimum conditions for electropolishing for each material. The ratio of the phosphoric acid and sulfuric acid was changed to determine the optimum composition for each material. It may be noted that the voltage across the electrodes was varied during the process of optimization. It was found that, the composition reported by Zamin *et al.* [16] was unsuitable for Mo sheets. The blue patches of molybdenum trioxide developed after electropolishing Mo was attributed to the oxidizing action of the sulfuric acid and hence the electropolishing was carried in orthophosphoric acid only.

Table 1. Optimum condition for electropolishing

| Parameter | Molybdenum | Inconel | SS |
|-------------|--------------------------------------|---|---|
| Electrolyte | H ₃ PO ₄ (85%) | H ₃ PO ₄ (85%) and H ₂ SO ₄ (5:1) | H ₃ PO ₄ (85%) and H ₂ SO ₄ (4:1) |
| Voltage, V | 7 | 6 | 6 |
| Time, min | 6-10 | 5-8 | 5-8 |
| Observation | Shining surface | Shining surface | Shining surface |

Condition after electropolishing

It is observed that the micrographs of the electropolished heat shields show improved surface condition of all the three types of samples. Figure 6 presents a comparison of the micrographs showing the improvement caused by the exercise.

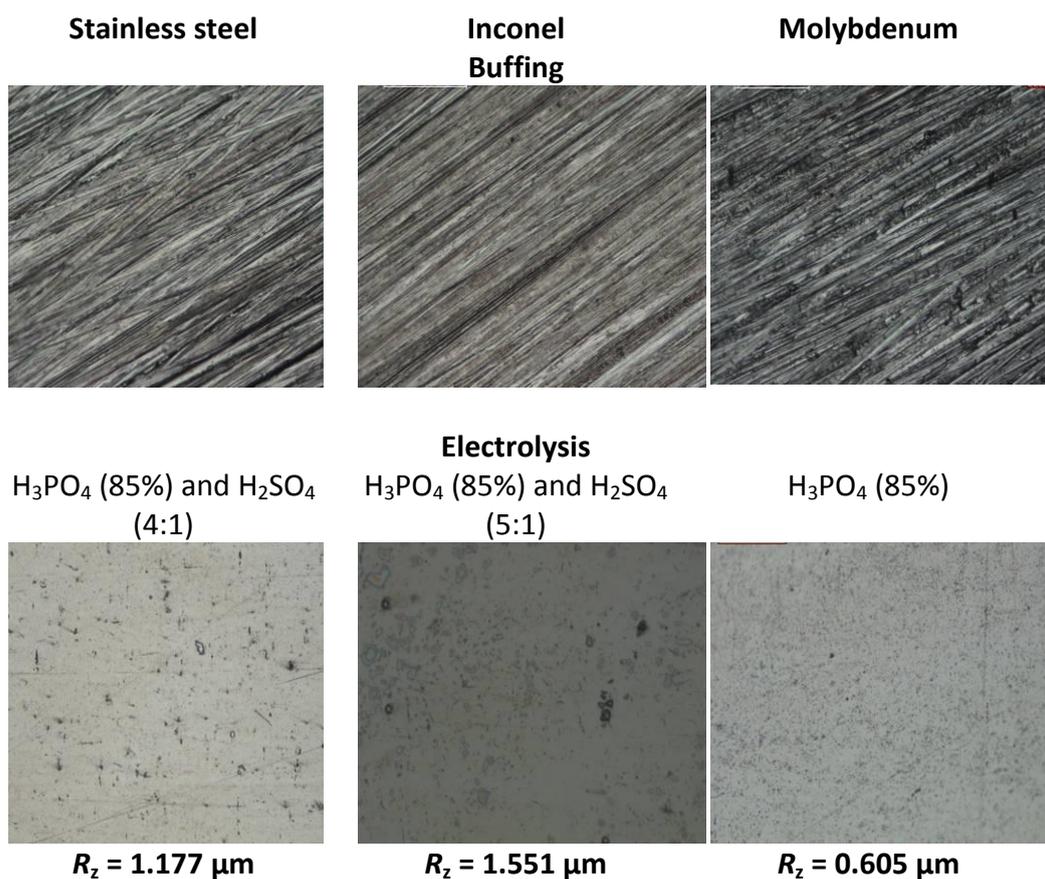


Figure 6. Micrographs of the electropolished heat-shields

Improvements in emissivity and roughness

As discussed earlier, emissivity of the surface is the most important parameter governing the performance of the shields. Since the roughness of the surface affects the emissivity values, an improvement of the surface profile was observed after the exercise. Table 2 gives the values of the surface roughness while Table 3 summarizes the improvement in emissivity caused by electropolishing. The roughness values of the buffed samples before and after electropolishing were compared to determine the etch rates. The etch rate of molybdenum was found to be the fastest and that of SS was the slowest in their respective electrolytic baths.

This encouraged us to carry out similar polishing on the large shields used in the industrial furnace. The dimensions of the large heat shields vary from 500×500×1 mm to 2000×2000×1 mm.

Figure 7 shows the refurbished heat shields. The heat-shields are cleaned and polished to a roughness less than $2\ \mu\text{m}$. The effect of electropolishing was demonstrated in the performance of the heat shield in the subsequent heater test. The graphite structure was found to achieve the desired temperature at a much lower power compared to the previous test due to the improvement in the emissivity values (Table 4). Hence the objective of the refurbishment of the heat shields was fulfilled.

Table 2. Roughness values of the samples

| Specimen | Roughness before electro-polishing | Roughness after electro-polishing | Reduction in roughness R_{z1} / R_{z2} | Etch rate, (mg/cm^2) / min |
|----------|------------------------------------|-----------------------------------|---|---|
| | $R_{z2} / \mu\text{m}$ | $R_{z2} / \mu\text{m}$ | | |
| SS-316 | 10.410 | 1.177 | 8.84 | 1.054 |
| Inconel | 8.162 | 1.551 | 5.26 | 1.116 |
| Mo | 9.380 | 0.605 | 15.5 | 1.800 |

Table 3. Emissivity values of the samples before and after refurbishment

| Material | Emissivity at $0.65\ \mu\text{m}$ | |
|----------|-----------------------------------|-------|
| | Before | After |
| SS-304 | 0.82 | 0.31 |
| Inconel | 0.78 | 0.5 |
| Mo | 0.7 | 0.27 |

Table 4. Heater power requirement

| Parameter | Heat shield before refurbishment | | Heat shield after refurbishment | |
|----------------------------|----------------------------------|------|---------------------------------|------|
| | Heater power, kW | 124 | 320 | 124 |
| Temperature of graphite, K | 1200 | 1523 | 1346 | 1546 |

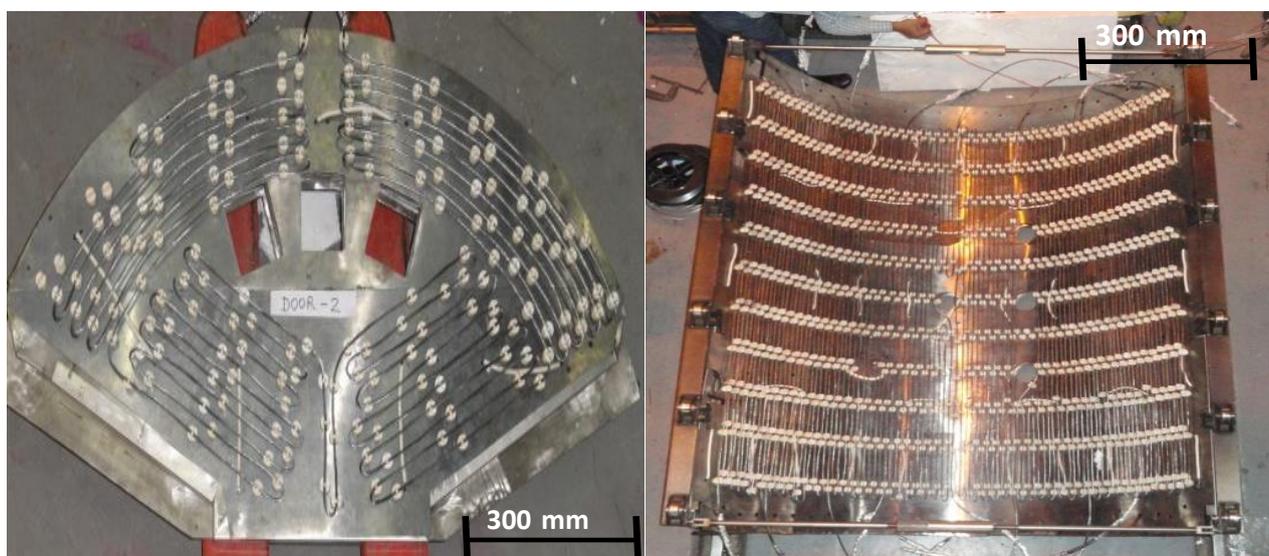


Figure 7. Electropolished heat shields

Conclusion

The present study gives a simple route for electropolishing of Mo and Inconel on a large scale using the commonly available laboratory chemicals. The results obtained in the trials were further

analyzed and the roughness and emissivity of the surface was measured to qualify the polish. From Table 2, it is evident that the reduction in roughness has been the highest for Mo, followed by SS and Inconel. A good improvement of emissivity of the shields was observed. Since surface roughness, among other factors, influences the emissivity of a material and catalysis the surface reactions, a reduction in power fed to the heaters.

Electropolishing of heat shields reduces the heater power requirement of the furnace and therefore plays a significant role in the design and economics of vacuum furnaces. The power requirement came down by 33 % from the earlier 320 to 210 kW.

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