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## On the effect of annealing on total normal emittance of oxidized steel

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### INTRODUCTION

It is known that the radiative properties of metals are intimately related to the surface conditions because the penetration of electromagnetic waves is limited to a very thin layer. Hence, surface damage related to polishing processes and roughness and oxide layer thickness are very important parameters affecting both total and spectral emittance [1–6]. Further it has been shown [7] that when a metallic surface undergoes an oxidation process, previous polishing and its particular technique can considerably affect the oxide layer growth and therefore the surface's emittance. As a consequence, radiative properties of oxidized metals may largely depend upon the 'history' of the sample before oxidation.

The aim of this note is to investigate the influence of annealing on total normal emittance of oxidized steel samples.

# EXPERIMENTAL RESULTS AND DISCUSSION

Total normal emittance tests have been carried out by the experimental set-up shown in Fig. 1 and previously described in ref. [8]. It allows a direct comparison of the total and spectral radiances issued by the surface under test and a blackbody cavity.

The two radiances are alternately focused onto the entrance slit of a grating monochromator by means of a system of mirrors  $L_1$ ,  $L_2$ ,  $L_3$ . A high-vacuum Hilger-Schwartz thermopile collects the radiation emitted by the sample surface and by a blackbody cavity. A P.A.R. lock-in amplifier is used to measure the thermopile output.

The size of the blackbody opening  $(1 \times 6 \text{ mm})$  and the cavity dimensions yield an estimated apparent emittance of 0.99. The temperature difference between the sample and the blackbody cavity is held within 1 K by means of an automatic temperature controlling system.

Before the test the experimental set-up was checked against spectral emittance standards (NBS). The difference between the measured and certified values fell within the standard deviations specified by NBS in the wavelength range 1-7  $\mu$ m.

Since the oxidation rate of a metallic surface is strongly affected by many parameters such as physico-chemical characteristics of the material, oxidation, temperature and surface status, it is very important, in order to study the influence of annealing, to control the surface finishing as much as possible.

In ref. [9] it was shown that the total normal emittance of this steel after oxidation in air was influenced by the initial surface roughness. An extremely low oxidation rate was observed on a polished mirror surface compared with that on surfaces with a greater initial roughness. On the other hand the same initial roughness does not involve a similar oxidation rate. In ref. [7] an increase of the average emittance by a factor of about 2.5 was observed when the sample surfaces were polished to the same final roughness by an industrial or a metallographic procedure. This effect, ascribed to surface damage can be removed by a suitable electrochemical polishing process.

In order to study the effect of annealing on the total normal emittance of oxidized steel, two series of ten samples each were obtained from a rolled-mill plate of AISI 316 L steel, and one of them was annealed at 1350 K in an argon atmosphere for 1 h.

All the specimens of the two series were then mirror polished

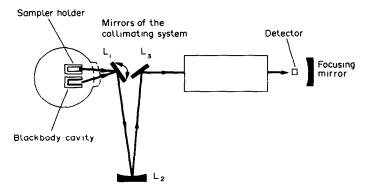


Fig. 1. Scheme of optical system.

310 Technical Notes

by the metallographic procedure used in ref. [10]. Before the emittance tests, all samples were oxidized in air at 900 K over periods up to 25 h.

At this temperature the oxidation rate of the surface is sufficiently low to allow the emittance to be correlated to the oxidation time with accuracy.

Emittance tests were carried out at the same temperature, to investigate the behaviour of the total normal emittance as a function of the oxidation time.

The experimental results are shown in Fig. 2 as average emittance of each set vs the oxidation time. The vertical bars show the variation in emittance obtained in each set of ten samples.

Figure 2 shows that the emittance  $\varepsilon$  depends on oxidation time and that higher  $\varepsilon$  values are observed for the unannealed samples. For both sets the emittance increases steeply with time during the first 6 h of oxidation, while thereafter stable emittance values are reached. In order to analyse the difference in the emittance values the surfaces of the two sets were microscopically observed.

Figure 3 shows typical magnified photographs of the surfaces of two samples of each set, oxidized at 900 K for 1 h.

The photographs indicate two different oxide layers which are both very thin. The crystalline grains of the annealed one are differently coloured due to the interference effects of light which occur in very thin oxide layers. In contrast the grains for the unannealed samples are colourless due to the oxide layers being thicker. In Fig. 3(a) the darker stripes show clearly the effect of the rolling-mill treatment.

At the beginning of the oxidation process, the lower  $\epsilon$  values of the annealed samples can therefore be abscribed to a thinner oxide layer than that of the unannealed ones.

At longer oxidation times the different stable emittance values are also due to different oxide thicknesses determined by a microscopical observation of sectioned samples. The average thicknesses were  $l\cong 1$  and 2  $\mu m$  for the annealed and unannealed samples, respectively, showing that emittance increases with an increase in oxide layer thickness, l.

In a previous paper [10] the influence of the oxide layer growth on emittance was investigated for the same steel oxidized in air at 1050 K over a period up to 3 h. The emittance was found to increase steeply with oxidation time during the first 30 min, to decrease slightly between 30 and 60 min and then to increase again at a much slower rate with increasing oxidation time. Such a behaviour was ascribed to an initial uniform growth of the oxide layer during the first 30 min, while at higher oxidation time, the slight variation of the emittance was ascribed to a surface modification due to a partial detachment of the oxide layer. At 1050 K a 0.44 $\varepsilon$  value was measured for an oxide layer thickness  $l \cong 3 \mu m$  and this result

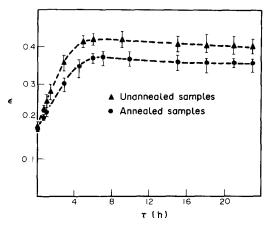


Fig. 2. Total normal emittance vs oxidation time  $\tau$  for the two sets of annealed and unannealed samples.

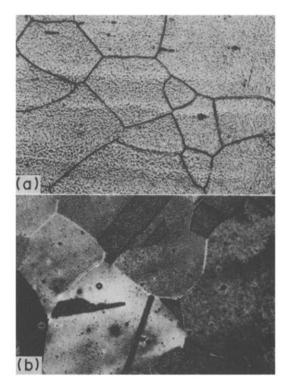


Fig. 3. Surface samples (×300) showing the different oxide layers after exposure to air at 900 K for 1 h: (a) unannealed; (b) annealed.

can be compared with the stable emittance value obtained here when the different test temperatures are taken into account.

A similar effect of the oxide layer thickness on total normal emittance has been observed in ref. [4] at a temperature of 400 K for the Al-Al<sub>2</sub>O<sub>3</sub> and Cu-CuO systems. For the former the authors found  $\varepsilon$  values to increase almost linearly from 0.05 to about 0.55, corresponding to an increase in oxide thickness up to 3.6  $\mu$ m; for the latter they found that the increase of emittance from 0.1 to 0.8, is linearly dependent upon oxide thickness from 1 to 5.5  $\mu$ m.

The low final  $\varepsilon$  values for both our sets mean that the resulting thin oxide layers largely hinder the oxidation process from going further. This can be ascribed to the protective effect of the preferential oxidation of chromium in iron—chromium—nickel steels [11]. Chromium tends to oxidize faster than iron and then chromium oxide becomes concentrated at the surface forming a thin protective layer through which the oxygen diffusion is less rapid than through iron oxide [12].

At the higher temperature (1050 K) the protective influence of chromium was less effective. Oxide layer thicknesses of about  $l \cong 30 \,\mu\text{m}$  were measured after 30 min of oxidation [10].

Finally, during annealing in an argon atmosphere, possible evaporation of chromium from the surface could modify sample composition depending on its diffusion rate which is rather rapid at 1350 K. The subsequent polishing and surface treatment could expose a chromium-deficient surface. Such an alteration of chromium concentration might lower the protective effect of chromium, increasing the oxidation rate and therefore the emittance [13]. On the other hand, as it is generally known, the annealing process reduces the oxidation rate because it removes the internal stresses caused by the rolling mill.

In conclusion, in the present experimental conditions, where preferential oxidation occurred, the different thickness of the two oxide layers can be explained by the effect of annealing on the rate of growth of the oxide layer.

The annealing process reduces the emittance of the oxidized samples because it decreases the oxidation rate. The lower

Technical Notes 311

oxidation rate of annealed samples can be ascribed to the removal of internal stress caused by the rolling mill. There is no indication that protective oxidation was reduced by annealing. At higher temperatures the effect of annealing on emittance may be less significant due to thicker oxide layers in the absence of protective oxidation.

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## Corrigenda et addenda to two-component Bénard convection in cylinders

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In a RECENT paper [1] Crespo and Velarde present a linear stability analysis of Bénard convection in a nonreactive binary (gas or liquid) mixture in a vertical cylinder heated from below. Both asymmetric and axisymmetric convective patterns are considered although the main object of the paper refers to the former and its relevance to describe recent experimental data by Abernathey and Rosenberger [2]. In this Note we correct some misprints that slipped through while correcting the galley proofs and some numbers that unfortunately occurred in the final formula for axisymmetric convection thus leading to numerical errors in a Table of results. Notation and equations refer to the paper by Crespo and Velarde [1].

In equation (1.2) the term  $SRa\Omega$  should be  $SRa\Omega$ . In equation (1.3) the term  $D_F\nabla^2\Omega$  should have a minus sign. In equation (1.4) the bracket  $(1+D_F)$  should have a plus sign. In Fig. 3 it should read  $D_F = -S^2/10$  (with a minus sign). In equation (4.2) the expression  $\partial [(1/r)\partial(ru)]/\partial r$  should be  $\partial [(1/r)\partial(ru)/\partial r]/\partial r$ . In equation (4.5) the factor S in front of the bracket in the fourth term should instead be  $r_D$ . In Fig. 5 the ordinate is  $Ra^c$ , as in Fig. 4.

In equation (4.11) some extra numbers appeared that unfortunately led to errors in the results reported in Table 1. The correct expression is

$$Ra^{c} = \{(200f^{2}/7\alpha^{2})[(1-\alpha^{2}/2)/f^{2}-21]$$

$$\times [5D_{F}\alpha^{4}/f^{4}-6(\alpha^{2}/f^{2}+10)\alpha^{2}/f^{2}]$$

$$+6(\alpha^{2}/f^{2}+10)^{2}\alpha^{2}/f^{2}$$

$$-5D_{F}(\alpha^{2}/f^{2}+10)\alpha^{4}/f^{4}\}$$

$$\times [6\alpha^{2}/f^{2}+5S(\alpha^{2}/f^{2}+10)/r_{D}$$

$$-5S\alpha^{2}/f^{2}-5D_{F}\alpha^{2}/r_{D}f^{2}]^{-1}.$$
(4.11)

Table 1. Number of axisymmetric rolls as a function of aspect ratio f = R/L. Large values of f approximate the infinitely extended horizontal layer. Note that for given aspect ratio the Soret effect induces a change of wavenumber. Results correspond to case  $r_D = 0.01$ 

| S<br>f | 0    |       | 0.01  |       | 0.1 |       |
|--------|------|-------|-------|-------|-----|-------|
|        | Rac  | Rolls | Rac   | Rolls | Rac | Rolls |
| 1      | 2975 | 1     | 1.418 | 1     | 248 | 1     |
| 2      | 1933 | 1     | 662   | 1     | 96  | 1     |
| 4      | 1903 | 3     | 638   | 2     | 76  | 1     |
| 7      | 1894 | 6     | 639   | 4     | 73  | 1     |
| 10     | 1889 | 9     | 639   | 6     | 73  | 1     |
| 100    | 1884 | 102   | 639   | 63    | 72  | 1     |

Note that, as in ref. [1], equation (4.11) accounts for both Soret and Dufour effects. The latter effect is disregarded in the following.

Corrections to equation (4.11) lead to corrections in Table 1. The correct and enlarged Table 1 is here given. Inspection of Table 1 shows that with a single trial function and a rather rough Galerkin calculation we obtain the one-roll structure in axisymmetric convection at S=0.1. The case f=100 is definitely a perfect approximation to the infinitely extended horizontal layer heated from below.

Although all possible applications are implicit in the equation (4.11) a straightforward analysis can help clarifying issues in some asymptotic limits. For instance, if we restrict consideration to the case of arbitrarily large aspect ratio the following results hold: