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# Iron oxide coating films in soda-lime glass by triboadhesion †

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**Abstract**

In the triboadhesion process the coating material is passed through a rotating cotton mop and the substrate to be coated. The cotton mop rotates at high velocity and exerts pressure on the surface of the substrate. The combined effect of pressure and velocity of the coating mop on the substrate increases its temperature close to the melting point, allowing deposition and diffusion of the coating material within the substrate. After it is deposited, its particles are embedded within the base material forming a thin film composite. The amount of the coating material deposited on the substrate has its maximum at the surface and then decreases as a function of the local temperature within the base material. Bearing this in mind, in the present work, triboadhesion is employed to deposit iron oxide in a substrate of soda-lime glass, with the purpose of determining the feasibility of using this technique for solar control coatings. It was found, through electronic scan microscopy, that a composite material film is formed following the coating direction. Reflectance and transmittance tests were carried out on the glass samples. A 20% difference was found in the visible spectral region (VIS), and a reduction between 10 and 20% in the Near Infrared Region (NIR). These results showed that the triboadhesion is a promising technique for the application of thin films for solar control or solar cells.

*Keywords*: Triboadhesion; Iron oxide; Soda-lime glass; Solar control; Light scatter

## 1. Introduction

Iron oxide, which is abundant in the earth's lithosphere, can be synthesized in pure or mixed oxides as well as doped structures. Iron oxide as described by Desai et al. [1] can be used as an electrode in non-aqueous and alkaline batteries. It also has potential applications in optical computing; in particular, because of its magnetic properties these oxides offer the possibility of high-density recording. Akl [2] describes iron oxide as a promising gas-sensing material. He also mentions that films of α-$Fe_2O_3$ may be obtained by several methods, such as chemical vapor deposition, sol–gel processes, pulsed laser evaporation, sputtering, hydrothermal technique and spray pyrolysis, among others.

On the other hand, efficient use of energy in buildings has provoked a growing interest in developing solar control thin films. These have the ability to decrease the amount of energy that crosses through them, reducing by this means the thermal load inside a room, and in consequence the need for air conditioning. Among the most used processes for metal or semiconductors coating for solar control, because of its simplicity and low cost, is the deposition by chemical bath. Nair et al. [3-7] developed several semi-conductive thin films by chemical bath for solar control and solar cells. Nair showed that 2 to 6 hours are required to obtain a film of a 100 nm thick. In comparison, when pyrolysis is employed, approximately 40 min [2] is needed to deposit a film of 472 to 634 nm thick. This condition has limited the industrial production of glass with solar control.

Friction, as well as vibration, is not always desir-

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able. However, as described by Rodríguez-Lelis [8, 9], friction is employed to raise the temperature of the surface to be coated, up to the point where diffusion may be achieved. Then, the coating material is introduced forming a composite material. This technique was applied to coat ball and tilting pad bearings with DLC [10, 11]. It was found that wear rates can be decreased up to 300 %, when the triboadhesion coating is applied to the inner running path of ball bearings. Also, slip could be induced as a result of the low friction factor achieved, when coating is carried out on the journal of tilting path bearings. In the triboadhesion coating process, it is claimed that it does not require a previous treatment to produce the coating, and because of its characteristics, this is carried out instantly. Rodriguez-Lelis [9] also claims that almost any coating material can be coated on almost any surface. This work attempts to demonstrate the feasibility of using the triboadhesion process to generate iron oxide film coatings on glass.

## 2. The deposition

As already described, the deposition of the iron oxide on soda glass was carried out by triboadhesion. A schematic diagram for the deposition process is shown in Fig. 1. This process is composed of 1) rotating wheel system, 2) force measurement system, 3) feeding system and 4) data acquisition system. In the deposition process, the cotton mop (c) rotates at high velocity and is pressed against the substrate to be coated, which was previously placed on the base (e). The base and force measurement system has a longitudinal movement that limits the time that the cotton mop and substrate remain in contact. Once the cotton mop and substrate are in contact, the cotton strings rub against the substrate surface and the temperature rises in an area of the order of the cotton strings thickness. This temperature rise can be calculated as described by Stolarski [12], among others.

This technology takes advantage of the heat generated by friction, that is, voids are formed and disrupted through statistical fluctuations caused by the temperature increment. Thus, the steady state population of voids may be expressed by:

$$N_C = N \exp\left[-\frac{G_T^*}{K T}\right] \tag{1}$$

where T refers to temperature, N and Nc are the number of atoms and cavities per volume, K is the Boltz-

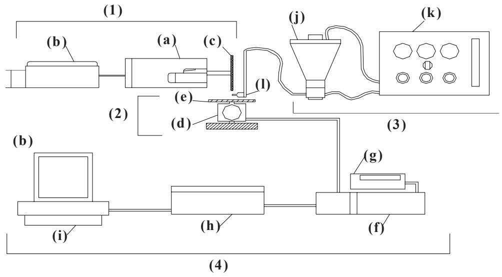



Fig. 1. Deposition process: 1. Rotating wheel system: (a) high speed motor, (b) velocity control, (c) mop; 2. Force measurement system: (d) ring type load cell, (e) base; 3. Feeding system: (j) particles container, (k) pneumatic control system, (l) nozzle; 4. Data acquisition system: (f) amplifier, (g) volt-meter, (h) signal analyzer, and (i) PC [6].

Fig. 2. A carbon mapping on copper obtained by SEM.

mann constant, and $G^*_T$ the free energy, which can be obtained as shown in [8]. From the free energy can be obtained the cavity critical radius, and from Eq. (1) the population of cavities where a particle of the coating material is likely to be introduced. If the temperature is high enough to promote diffusion, the particles will travel within the coated material forming a thin composite film [8, 9]. These characteristics will produce a film coating, which will have the highest concentration at the surface, where the maximum temperature is achieved. This concentration decreases as a function of the local temperature within the base material. An example of the final distribution obtained with the triboadhesion process is presented in Fig. 2. This figure shows a cross section mapping of a copper probe coated with DLC by triboadhesion, where it is noted how the concentration of particles varies from top to bottom.

## 3. Experimental details

### *3.1 Triboadhesion coating*

Before carrying out the coatings, as described in [8], the applied load and temperature had to be determined for the specific rotating velocity, which for the present work was 28000 rpm. The temperatures theoretically evaluated showed that these could vary from 700 $^{o}$C up to 1270 $^{o}$C at 0.1 N and 1 N, respectively. The force, mass flow, rotational velocity and tests carried out in this work are shown in Table 1. The substrates employed for the deposition were glass rectangular strips 2.5 cm wide, 5 cm long and 3 mm thick. In the present work, rotational speed and the mass flow were kept constant during the whole test, and only the number of times that the mop passed through the coating surface was varied, as described in Table 1.

Once the coating was carried out, each probe was rinsed abundantly with water to avoid any iron oxide residue. One of the probes, coated with iron oxide, is shown in Fig. 3. In this probe, a central dark area may be distinguished from the bulk of the glass probe, and it is identified as the coating street formed by the deposition. In a previous numerical heat transfer analysis, it was found that the thickness of the coating film is close to 6 microns. Based on this result, the analysis was restricted to morphologic and optical measurements.

### *3.2 Optical characteristics*

For the optical tests, a spectrophotometer Shimadzu 3100 PC UV-VIS-NIR, with an operation range of 250 to 2500 nm, was employed. As already described the cotton mop strings rub against the substrate surface. This interaction itself may modify the surface roughness and the optical characteristics of the soda lime glass.

Thus, in order to evaluate the effect, on the optical properties caused by of the coating mop strings colliding on the surface, three tests were carried out. For these tests, a 3mm soda-lime glass was employed and no coating material was introduced. The number of times that the cotton mop passed through the glass was the following: for the first probe, 3 times; for the second, 5; and for the last one, 8 times. These three tests were carried out at the same velocity and load. The transmittance tests for these probes are shown in Fig. 4. It can be seen from this figure, that the three plots are superimposed on one another following exactly the same path, thus showing that there is not any influence on the optical characteristics caused by the mop passing through the surface, nor on the surface characteristics.

Once the influence of the interaction of the cotton mop on the substrate surface was evaluated, samples with iron oxide were prepared. As shown in Table 1, three tests are presented in this work. These differ only in the number of passes of the cotton mop through the surface. The transmittance graphs for these tests are shown in Fig. 5(a) and the reflectance graphs in Fig. 5(b). Both plots of figure 5 are divided in three sections.

Table 1. Test parameters to coating soda-lime glass samples by triboadhesion.

| T | Feeding Material | $F_{av}$ (N) | □ | Rot. Speed (rpm) | Mass Flow (kg/min) |
|---|---|---|---|---|---|
| a | Iron oxide | 0.8±0.0816 | 3 | 28,000 | 1.5 |
| b | Iron oxide | 0.8±0.0816 | 5 | 28,000 | 1.5 |
| c | Iron oxide | 0.8±0.0816 | 8 | 28,000 | 1.5 |

T= Test number, Fav=average force, δ= How many times the mop pass through the surface

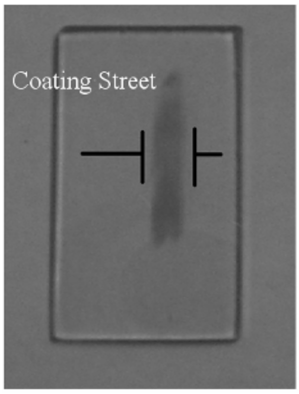



Fig. 3. Soda-lime glass sample coated with iron oxide by triboadhesion.

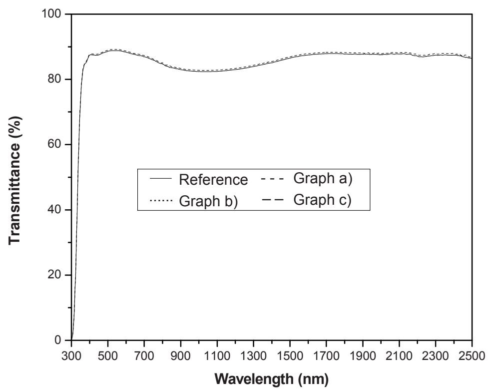
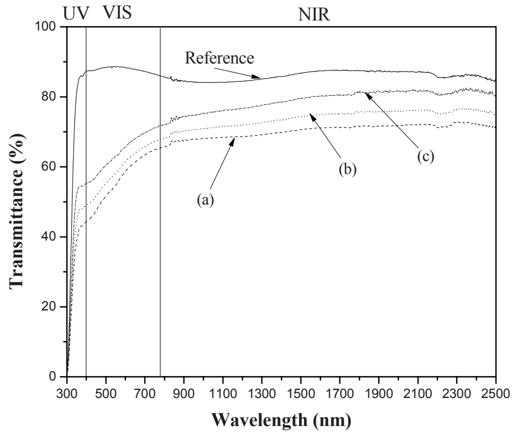
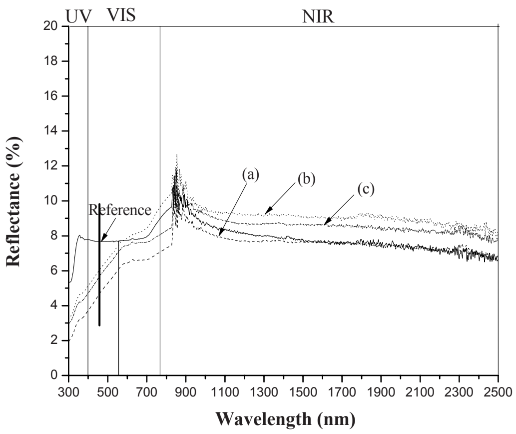



Fig. 4. Transmittance results of the test on soda-lime glass without feeding material.




(a)




(b)

Fig. 5. Triboadhesion test results on a soda-lime glass sample coated with iron oxide by triboadhesion: a) Optical transmittance and b) reflectance. The terms UV, VIS and NIR represent the Ultra Violet, Visible and Near Infrared spectral radiation regions.

The first is the ultra violet spectral region (UV), which varies from 300 to 370 nm. The second, the visible spectral region (VIS), with a span from 370 up to 790 nm; and the last one, near infrared spectral region (NIR), that goes from 790 up to 2500 nm. In these graphs, a general reduction of the transmittance close to 20 % is appreciated with respect to the reference. However, it should be pointed out, that in the VIS spectral region, the steep slope shown by the coated samples, the variation on transmittance reduction varies from 49% up to 21 %. Also, the transmittance decreases as the number of times the mop passes through the surface increases. The reflectance exhibits a similar behavior, where the average of all thin films is less than 10 % in the solar spectral region. As an interesting feature, the test (b), after 500 nm, shows a higher reflectance while tests (a) and (c) remain below the reference.

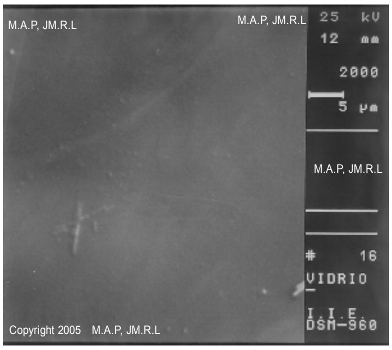
The singular behavior, appreciated in the visible spectral region (VIS), was attributed to the anisotropy of the thin film. These results can be explained considering the amount of heat gained by the base material during friction. Since for very high loads and velocities the heat gained by the glass was too large, provoking the probes to break, a compromise between load and velocity had to be made, reducing the deposition temperature and in consequence the amount of material to be deposited. Then, at three passes, the material deposited on the surface was less than at five or eight, being the one with eight passes the more continuous film. However, no assurance could be made regarding the scattering of the particles deposited.

As already shown in Fig. 2, the coating by triboadhesion coating process produces a thin composite material. Thus, it was envisaged that the scattering [13-15] of the coating particles has an influence on the amount of the light passing through the glass. If one could consider a lineal behavior, the reflectance and transmittance would have varied as a function of the number of passes; however, probe (b) exhibited a different behavior on the reflectance measurements, indicating that random distribution and size of particles has an influence on the results. Hence, none of the probes will ever be equal to another.

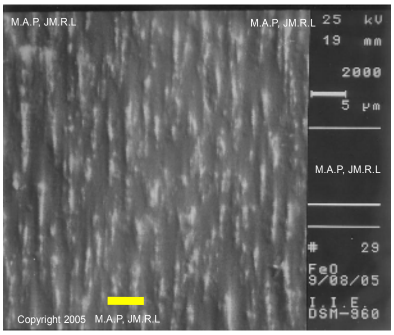
### *3.3 Scanning electronic microscope (SEM) analysis.*

In order to estimate the final surface texture of the coated samples, SEM analyses were carried out. A ZEISS-960 scan microscope was employed for these tests and the magnification used was 2000X. In Fig. 6 are shown micrographs of the surface (6a) without and (6b) with coating. It may be seen, from Fig. 6b, that although particles between 1 and 60 microns were fed, smaller particles can be distinguished. It can also be noted, that valleys and mountains are formed in the direction of the coating process.

It may also be seen that particles are partially embedded in the surface of the substrate. From here, it was thought, following Rabinowicz [16] and Tabor [17], that the smaller the particles fed the higher the adhesion force, i.e., that for small particles the probability of adhering to the surface increases.




(a)




(b)

Fig. 6. SEM micrographs for the soda-lime glass samples: (a) without coating (b) with iron oxide coating.

## 4. Conclusion

The first results obtained from triboadhesion deposition process of iron oxide on soda-lime glass are presented. A general reduction in transmittance patterns of approximately 20% and a 10% variation on the reflectance patterns was found. In the NIR region, the path followed by the measurements in all probes is similar. In the visible spectral region a steep behavior is noted, which was attributed to the scattering of particles and its influence on the transmittance and reflectance results. From this, considering the scatter of particles of the composite thin film coating produced by the triboadhesion, its potential for solar control by enhancing reflectance or transmittance as a function of the order of scattering may be foreseen, that is, there is a relation between the geometrical distribution of particles and the ability to increase or reduce transmittance and reflectance.

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