

# Comparison of material structures and scratch strength of thin films of glass mirrors and automobile windscreens

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**Abstract.** Several types of European and Asian automobile windscreens coated with single and multilayer fixing fluxes and silver thin films were studied, and their material structures and their scratch properties were compared with glass mirrors' by the authors. The realized laboratory tests had shown very strong influences of cracks and bubbles in the microstructures of fixing thin film layers and silver coatings on scratch resistance, acoustic emission and micro-hardness of the coatings both on glass mirrors and automobile windscreens. To examine the material structure of flux thin film layers and silver coatings scanning electron microscopy (SEM) was used. The scratch resistance, acoustic emission, nucleation and propagation of cracks were studied by an optical digital mechanical scratch tester.

## 1. Introduction

The aesthetic appearances and quality of glass mirrors strongly depend on material structures, quality and thickness of fixing flux thin film layers and silver film coatings used. Parallel to this, in automobile industry the efficiency of windscreen heating also depends on materials used and on the microstructures of fixing thin film coatings, heater filaments and defects occurring at the interfaces between themselves and the glasses. To evaluate the adhesive strength of coatings at the interfaces of ceramic, glass, plastic or metallic substrates scratch test is a widespread technique. Using spherical and conical diamond indenters a series of single scratch test was performed on soda-lime glass by Shapiro and Li [1], meanwhile De Rosa and Wagner [2] examined the scratch resistance of polyimide coatings of different Young's modulus for alumina-silicate glass surfaces. In their works Puskas [3] and Gömze [4] studied the scratch resistance of different ceramic and porcelain glazes, and determined their micro-hardness as well. Investigating of glazes and glazing processes used for ceramic roof-tile industry Puskas, Geber and Gömze [5] have found traceable relationship between frost resistance and durability of roof-tiles and scratch strength of their glazes. Using finite element methods Bull and Berasetegui [6] gave an overview and quantitative evaluation of coating adhesion measurement by scratch testing. Kataria, Kumar, Dash and Tyagi [7] had shown that the results obtained from the scratch test depend on number of parameters, like loading rate, surface roughness, scratch speed and material properties. Using single-pass scratch test and finite element simulation Subhash and Zhang [8] could successfully model the surface roughness of coatings. The role of friction in material characterization via indentation scratch tests is still a challenging field for researchers in applied mechanics like Hashiguchi and Ozaki [9] or physicians like Fusco, Smith, Urbakh and Vanossi [10]



and like Luo et al. [11]. The influence of material structures as well as bubbles and cracks on surfaces and inside of coatings on their scratch resistance were studied by Xie et al. [12].

There are several methods to test adhesive strength and material structures of fixing flux thin film layers, silver films, heater filaments and the interfaces between themselves and the glass substrates, but scanning electron microscopy and scratch test are the most common. Zaidi, Djamai, Chin and Mathia [13] successfully characterized the adherence of diamond like carbon (DLC) coatings through scratch test. Their instrument was equipped with sensors for normal and tangential forces and an acoustic detector to detect the nucleation and the propagation of cracks. The adhesive strength and scratch resistance of hybrid organic-inorganic coatings on float glasses were tested and reported by Malzbender and de With [14]. They found that the main reason for delamination was the deflection of cracks at the interfaces. Scratch test method and scratch resistance analysis were done by Boentoro, Pflug and Szyszka [15], when a high mechanical stability was required against optical coatings on glasses and polycarbonates used in automotive and architectural purposes. At the same time the efficiency of light transmission of float glasses considerably depends on containment proportions of FeO and Fe<sub>2</sub>O<sub>3</sub> in glasses as it was underlined by Paroczai and Erdelyi [16]. To examine the nano- and microstructures of different types of materials including thin film layers and coatings the scanning electron microscopy (SEM) test is the most widespread method. A SEM test was used by Kulkov and Savchenko [17] also to identify the deformations in material structure at the surface and near the surface of zirconia based ceramics under high-speed dry sliding on steel; and by Ershova and Kelina [18] during development high-temperature wear-resistant bearings based on silicon nitride. SEM tests were also used by Hernandez, Torre and Rocha-Rangel [19] during synthesis and microstructural analysis of alumina-matrix cermets, and by Katahira and Ohmori [20] to examine and evaluate the nano-level surface roughness.

Since the 1980's there are several patents to increase the efficiency of the silver coatings on glass mirrors. Tracy and Benson [21] used silicon nitride coating to protect silver layers on glass mirrors. Ishikawa et al. [22] have developed a special device including a securing element affixed between the back surface and the mirror cover plate for reducing deformation of said mirror. Fujii [23] developed a special surface reflecting multilayer film to increase the surface reflecting properties of mirrors. Park [24] was one of the first scientists who developed coating system transparent to light and reflective to thermal radiation. At present there are several patents to increase efficiency of both silver coatings on glass mirrors and the fixing of heating filaments of automobile windscreens.

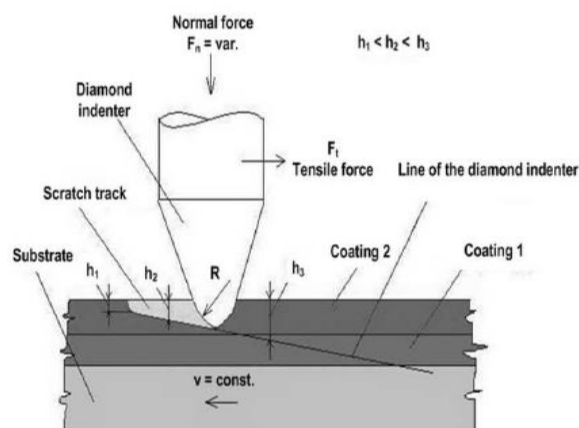
The consequences of defects in the micro- and nanostructures of fixing thin film coating layers or heater filaments and glasses at interfaces considerably reduce the efficiency of both glass mirrors and windscreen heating. Because of the strong reduction in efficiency of light reflection of mirrors and heating reduction of the automobile windscreens tested by authors, the aims of this work are the followings:

- open the typical defects taking place in material structures of flux thin film layers, silver coatings and heating filaments on automobile windscreens and glass mirrors;
- compare the scratch strength, acoustic emission and propagation of cracks inside the thin film silver coatings of glass mirrors and copper heating filaments of automobile windscreens and their interfaces with float glass surfaces.

## 2. Experimental

There are several methods to test adhesive strength and material structures of fixing flux thin film layers, silver films, heater filaments and interfaces among themselves and the glass substrates. To examine the nano- and microstructures of different kind of materials the scanning electron microscopy (SEM) test is the most widespread method. SEM can be used to examine and evaluate the nano-level surface roughness, or to reveal the influence of material micro- and nanostructure on mechanical properties. To characterize and give quantitative results for the mechanical resistance of the interfaces between the coating film layers, and between coating and the material on which it is deposited, scratch test method is the most applicable.

The material structures were examined by scanning electron microscopy and the adhesive strength of the thin flux film layers, silver coatings of glass, heater filaments and their interfaces with the copper clips and glass beds were determined by optical scratch tests. Commercial float glass mirrors and different automotive windscreens were used and examined on Hitachi TM-1000 SEM and Tear Coatings ST-3001 scratch tester. For scanning electron microscopy tests the specimens were 'cut out' from glass mirrors and windscreens having surfaces of  $15 \times 15 \text{ mm}^2$  and for optical scratch test by  $60 \times 30 \text{ mm}^2$ . The principle of the used optical scratch tester ST- 3001 is shown in figure 1.



**Figure 1.** The principle of optical scratch test

The scratch tester is equipped with sensors for normal and tangential forces, and an acoustic detector to visualize the nucleation and propagation of cracks and crack processes. The system is also equipped with an optical microscope to observe each event on the scratch. Single-pass scratch test was used with the following parameters:

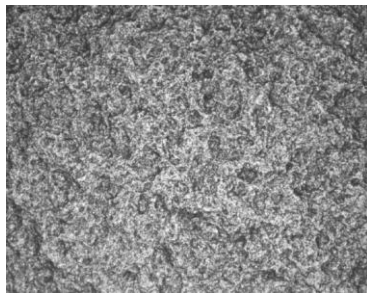
- Initial load: 3 N,
- Final load: 35 N,
- Loading rate: 50 N/min,
- Pause: 1 s,
- Unloading rate: 50 N/min,
- Linear displacement: 10 mm,
- Linear velocity: 10 mm/min,
- Maximum friction force: 200 N,
- Maximum acoustic emission: 110 dB.

### 3. Results and Discussion

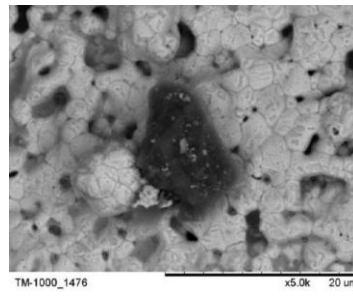
#### 3.1. Comparison of material structures of silver thin film coatings

During scanning electron microscopy several defects can be observed on the surfaces of silver thin films, fixing flux thin film layers and copper filaments. The typical surface roughness of the silver coatings of glass mirrors is shown in figure 2. The most characteristic defects in coatings of glass mirrors are: coarse roughness, deficiency and absence of material continuity including holes, inter-crystalline and trans-crystalline cracks, open pores and air bubbles.

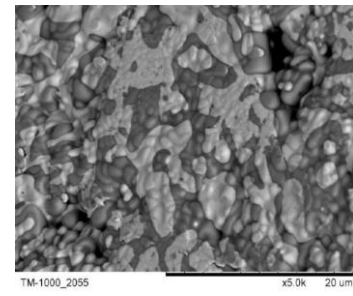
The typical surface structure of unpolished silver coating films on automobile windscreens is shown in figure 3. Because of the bad coating process and shortage of polishing the surfaces of the unpolished silver coatings on automobile windscreens are interrupted with craters, holes, humps and have many discontinuities. The same defects in material structures can be observed on surfaces of bedding flux coatings (figure 4), which reduce the efficiency of the bedding of copper clips and heating filaments into glass substrate of automotive windscreens.



**Figure 2.** Typical surface roughness of the silver coatings of glass mirrors

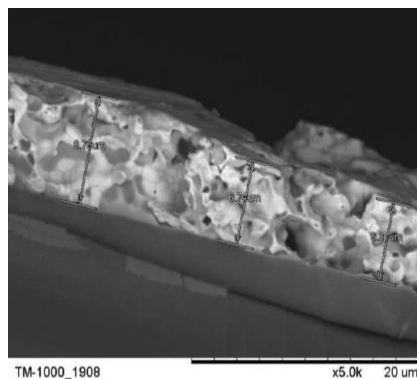


**Figure 3.** Surface structure of unpolished silver coating of automobile windscreens

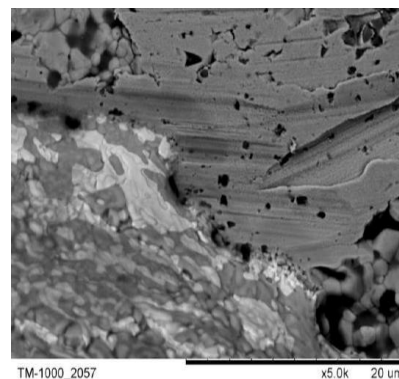


**Figure 4.** Typical surface structure of flux thin film layers

Because of the instability of the coating process and heat treatment the thicknesses of the final silver thin films do not have constant values, so pores and bubbles can be observed both at the glass surfaces of mirrors and automobile windscreens (figure 5). The defects in material structure and in cross-section of silver coating films, shown in figure 5, can be avoided using other bedding flux thin film materials, changing coating technology, temperature or heating times for post deposition heat treatments. The typical defects between the interfaces of fixing flux thin film layers and silver thin film coatings are shown in figure 6. There are several gaps, pores, bubbles and holes on the interfaces between the flux thin film layers and the polished silver coatings.



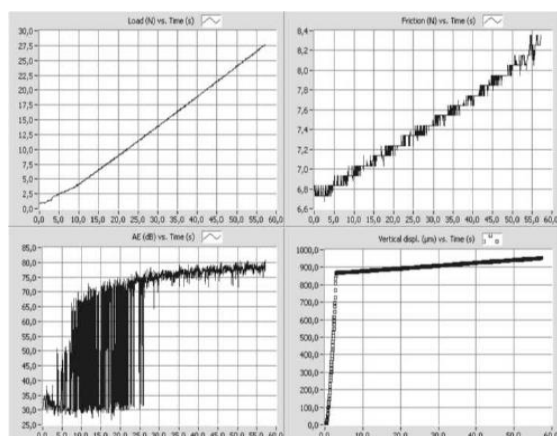
**Figure 5.** The typical defects of cross-section of the silver thin film coatings



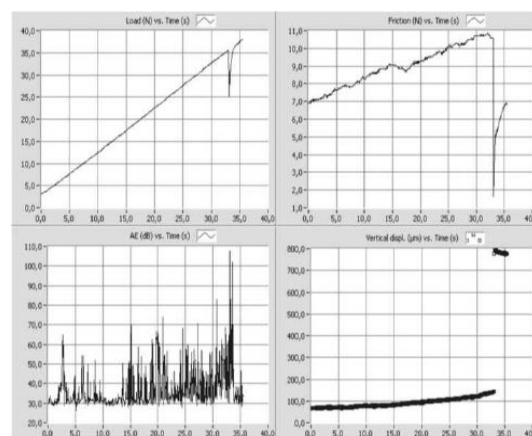
**Figure 6.** Some typical defects at the interfaces of silver coatings and flux thin film layers

### 3.2. Comparison of scratch tests of silver thin film coatings

A typical experimental results relating to the scratching of silver coatings on glass mirrors are shown in figure 7. The scratching curves give the evaluation of the normal load, friction force, acoustic emission and vertical displacement as a function of time. The rupture of silver coating thin film leads to a discrete acoustic emission with high amplitude and to the release of the stored elastic energy. At the beginning the acoustic emission is 30 dB and finally it achieves the value of 80 dB.



**Figure 7.** The typical scratching curves of silver coatings on glass mirrors



**Figure 8.** The typical scratching curves of silver coatings on glass of automobile windcreens

Scratch testing of thin film coatings on automotive windcreens revealed quite different behaviour (figure 8). Again, the curves show the evaluation of the normal load, friction force, acoustic emission and vertical displacement as a function of time. The rupture of silver coating thin film leads to a discrete value of 35 N of normal load, approximately 11 N of friction force and the acoustic emission with high amplitude from 30 dB to 107 dB and to the release of the stored elastic energy. The picture of a typical scratch track on surface of silver coating and copper heating filaments of automobile windscreen is shown in figure 9.



**Figure 9.** The typical scratch track

It is obvious from the scratch track shown in figure 9 that the copper filaments (in centre) are much wider than bedding silver coatings, which means the copper filaments are exceeded from the relative flat surfaces of coatings.

#### 4. Conclusion

Our investigation reveals many defects of the material structure of flux thin film layers and thin film silver coatings for commercial glass mirrors. The adhesion strength of silver coatings is much higher on automobile windcreens than on glass mirrors. This is obvious when we compare the normal load and friction force curves of figure 7 and figure 8. At the same time, during the scratch tests the maximum value of amplitude of acoustic emission is much higher at the automobile windscreen than for glass mirrors, which means higher level of elastic energy of deformation.

There are many defects in the material structure of the thin film silver coatings because of the instability of the used coating processes and heat treatment technology. There are also several defects at the interfaces of glass substrate, fixing flux thin film layers, and the silver coatings both at the glass

mirrors and the automotive windscreens because of the insufficient wetting and the ability to spread the fluxes used. The wetting properties could be considerably improved by increasing the heating time and/or temperature gradient in the heat treatment curves after coating processes.

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