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Nano-scale analysis of titanium dioxide fingerprint-development powders

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Abstract. Titanium dioxide based powders are regularly used in the development of latent fingerprints on dark surfaces. For analysis of prints on adhesive tapes, the titanium dioxide is suspended in a surfactant and used in the form of a small particle reagent (SPR). Analysis of commercially available products shows varying levels of effectiveness of print development, with some powders adhering to the background as well as the print. Scanning electron microscopy (SEM) images of prints developed with different powders show a range of levels of aggregation of particles.

Analytical transmission electron microscopy (TEM) of the fingerprint powder shows TiO_2 particles with a surrounding coating, tens of nanometres thick, consisting of Al and Si rich material. X ray photoelectron spectroscopy (XPS) is used to determine the composition and chemical state of the surface of the powders; with a penetration depth of approximately 10nm, this technique demonstrates differing Ti : Al : Si ratios and oxidation states between the surfaces of different powders. Levels of titanium detected with this technique demonstrate variation in the integrity of the surface coating. The thickness, integrity and composition of the Al/Si-based coating is related to the level of aggregation of TiO_2 particles and efficacy of print development.

1. Introduction

The use of fine, dry powders to develop latent fingerprints left after criminal activity has been well established for many years [1]. However, various types of surfaces, such as rough materials, fabrics, wetted materials and adhesives have limited suitability with this type of technique. Other techniques are available, such as Gentian Violet, but use of powders in suspension has been shown to produce consistently more reliable results [2] when studying prints on adhesive side of tapes. A number of commercially available products are available for this, however, Richardson's work [3] shows variations in efficacy of nominally similar powder suspensions available from different companies, relating to levels of background staining – the powder adhering to areas where there is no print. This work uses electron microscopy and complementary techniques to elucidate the structure, composition and efficacy of two different powders.

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2. Analysis

This work compares two powders, commercially available for developing latent prints on the adhesive side of adhesive tapes. Powders are obtained from Stan Chem International, currently recommended by HOSDB [4], and Sirchie Fingerprint Laboratories. For demonstration purposes we show effectiveness on one type of tape used by Richardson [3], though comparative effectiveness of print development does vary with type of adhesive tape studied [3, 4].

2.1 SEM of developed fingerprints

A finger print was developed with each powder, representing the first depletion of a series of latent images on the adhesive side of proprietary black insulating tape. The resulting images were mounted onto adhesive carbon tape and examined within a field emission scanning electron microscope equipped with an energy dispersive X-ray analyser. Electron images and X-ray spectra were recorded to show the dispersion of the titania particles.

2.2 Powder analysis

2.2.1 XPS For XPS measurements, an aliquot of each dry powder sample was mounted on to carbon-loaded pressure sensitive adhesive, attached to polished copper, and any excess powder removed. Mounted samples were examined within an Escalab VG X-ray photoelectron spectrometer (XPS), utilising aluminium and magnesium anodes and variable iris and detector apertures to examine different size areas on the surface; a small area size was selected to reduce the background signal. The XPS technique gathers compositional data from the surface of the sample and up to a depth of less than 10nm into the sample; the detection limit is approximately 0.5 at. % in this region. XPS spectra were collected in the energy range 300-0 eV with a step size of 0.2eV, collection time 200ms and were averaged over 10 scans.

2.2.2 TEM For analytical TEM examination, samples of each white powder were diluted with distilled water and dispersed onto carbon films covering copper microscope grids. The samples were examined within a JEOL 2000FX transmission electron microscope equipped with an energy dispersive X-ray (EDX) analyser. Representative micrographs were recorded to show the morphology of the particles and any coatings that may have been applied to the particles. X-ray spectra were recorded from the coatings to determine their elemental composition.

3. Results and Discussion

3.1 Developed fingerprints

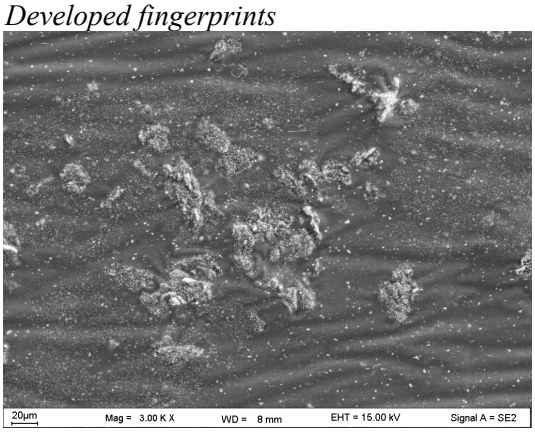

Figure 1. Sirchie showing aggregations and even dispersion of powder.

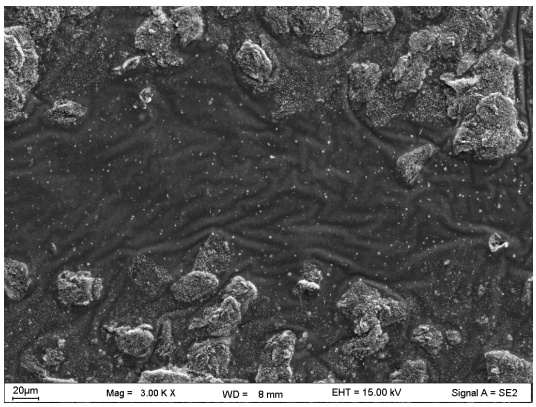

Figure 2. Stan Chem powder showing aggregates and defined demarcation of ridge areas.

SEM results show fingerprints on MT tape [3, 4] developed with Sirchie powder show aggregations of development particles with uniform background staining over fingerprint ridges and bare adhesive, figure 1. Stan Chem powder (figure 2), also shows aggregations, possibly related to skin cell removal, and also shows a greater staining of the ridge area and reduced staining of the background adhesive, providing ridge contrast in the developed image.

3.2 Fingerprint powders

The compositional data from the XPS spectra, as shown in table 1, indicates the presence of aluminium and silicon and low-to-negligible levels of titanium on the surface of each sample. The XPS technique examines only the top few nanometres of the sample surface, this signal therefore suggests that the TiO_2 powders are partially coated in a material rich in aluminium and silicon. The composition of this coating varies between powders, as highlighted in table 1 and the difference in levels of titanium observed suggest differences in the thickness or integrity of this coating.

Table 1. Comparative aluminium, silicon and titanium content of sample surfaces, calculated from analysis of XPS spectra

	Relative intensity / At. %		
	Ti	**Al**	**Si**
Stan Chem	9.6	61.8	28.5
Sirchie	-	40.5	59.5

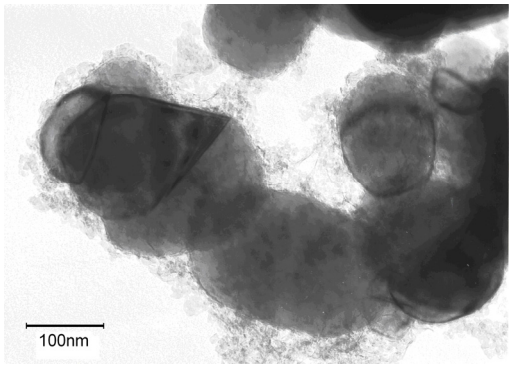

Figure 3. TEM images of Sirchie powder, showing TiO_2 particles and loose, thick coating.

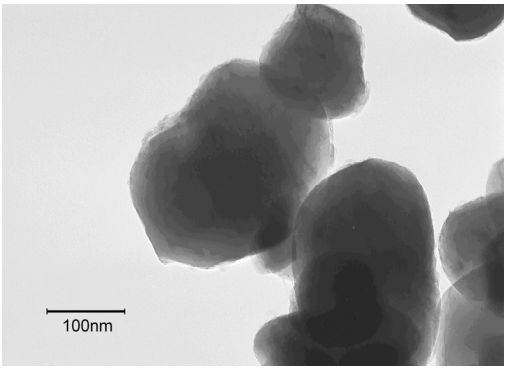

Figure 4. TEM images of StanChem powder, showing TiO_2 particles and discontinuous, thin coating.

Figures 3 and 4 are representative transmission electron micrographs of the powders showing the typical morphology of titanium dioxide. The micrographs also show that the powders have a coating of a diffuse nano-particulate nature, the thickness and elemental composition varying between the powders. X-ray analysis, figures 5 and 6, shows that the coating on both types of particles gave significant peaks for silicon and aluminium. The coating on Stan Chem has a higher relative concentration of aluminium than that on the Sirchie powder.

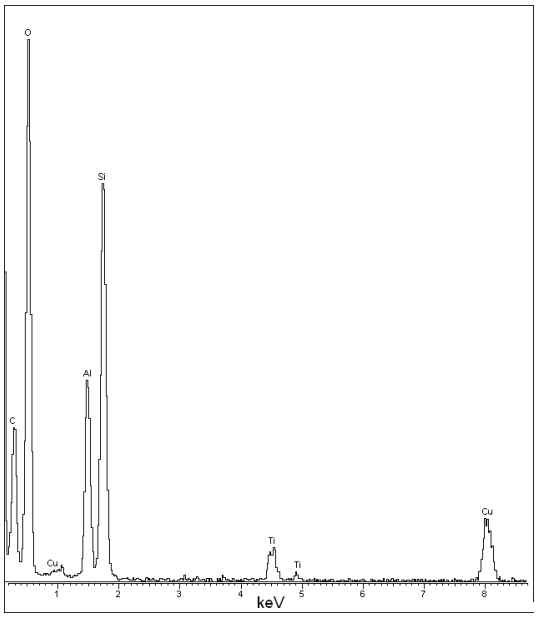

Figure 5. EDX spectrum from coating of Sirchie powder

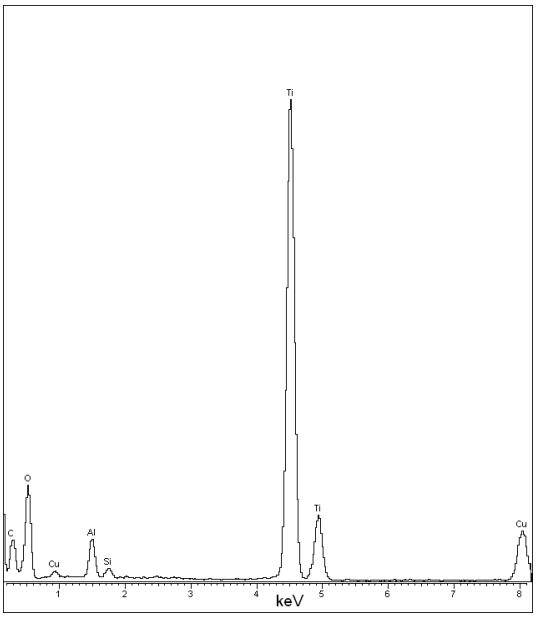

Figure 6. EDX spectrum from StanChem powder, showing strong Ti signal

Transmission Electron Microscopy images show the powders consist of TiO_2 particles with a surrounding coating either rich in Al or Si. The thickness, integrity and adherence of this coating vary substantially between the samples studied, as indicated in figures 3 and 4. Sirchie powder shows the thickest coating of a loosely spaced material, whereas in StanChem the coating material forms a discontinuous, thinner layer and is in some instances disassociated from the TiO_2 particles.

4. Conclusions

The two fingerprint powders examined gave differing performance when developing latent prints on black adhesive tape. Scanning electron microscopy (SEM) images of developed prints show various levels of aggregation of particles. X-ray photoelectron spectroscopy and analytical transmission electron microscopy of the powders indicate both powders consisted of TiO_2 particles approximately 300nm in diameter, with a coating that is rich in aluminium or silicon. In Sirchie powder this coating is loosely packed, and coats the TiO_2 particles to a thickness around 100nm; Stan Chem powder has a denser, thinner coating that irregularly covers the TiO_2 particles, reaching a thickness of 20nm in places. The presence and composition of this coating, and its adherence to the TiO_2 particles, may contribute to the cause of the varying performance of the powders in fingerprint development.

Acknowledgements

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