Assessing the Capability of Combining Elemental and Phase Mapping in Automotive Paint Systems Analysis Using SEM/EDS

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INTRODUCTION

After microscopical examination, scanning electron microscope/ energy dispersive Spectroscopy (SEM/EDS) is often coupled with Fourier Transform Infrared Spectroscopy (FTIR) in forensic paint analysis to obtain comprehensive chemistry regarding the composition of each coating layer[1,2]. FTIR is useful in detecting the organic binders; while SEM/EDS is excellent in gathering elemental details from the pigments and additives. Although standard elemental mapping with SEM/EDS Provides detailed elemental information, the indication of possible chemical compounds present in each layer is limited. Other than that, the overlapping of principal X-ray lines, such as that for Titanium (Ti) and Barium (Ba), cannot always be resolved by obtaining the elemental details alone.

The TruMap and PhaseMap analysis function on the Aztec software (Oxford Instruments, UK) are suggested to help resolve the overlaying issues and provide possible chemical constituents in each phase of the automobile paint systems respectively. In this project, elemental information of 13 automobile paint systems were collected using standard elemental maps, TruMaps, and PhaseMaps to evaluated and compare the performance of the new analysis methods to the conventional one. This work also investigated the capability of PhaseMap analysis in identifying possible chemical constituents and providing supplementary information On top of elemental mapping to determine if the new function can be used to provide investigative lead in forensic paint analysis.

MATERIALS & METHODS

Thirteen (13) automobile paint systems of known make, model and number of coating layers were obtained from the Texas Department of Public Safety, the Harris County Institute of Forensic Sciences' Quality Management Division, and Caliber Collision Center (3575 South Loop West, Houston, Texas). Samples of each paint system were secured with a metal clip and embedded in separate epoxy biscuits with the cross section side facing up (Figure 1). The surfaces of the epoxy biscuits were thoroughly polished to expose the cross sections of the coating layers.

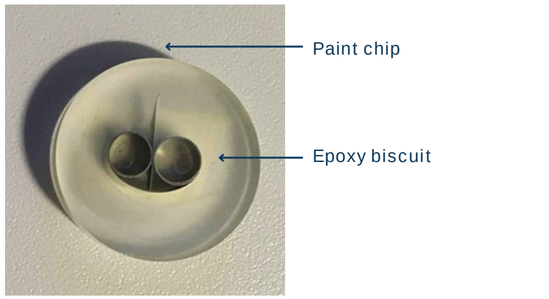

Figure 1. Epoxy biscuits containing paint chip samples

MATERIALS & METHODS (CONT'D)

The samples were subjected to analysis by a Hitachi SU 3500 (Tarrytown, NY) equipped with an Oxford X Max^N 50 X-ray detector (Concord, MA). Backscattered images of the cross sections were obtained and the electron images were first scanned for regular elemental data. Then, the same electron images were processed by the TruMap analysis to deconvolute any overlaying principle X-ray lines, if present, and the major contributing elements in each layer were listed. Possible compounds of the phases were evaluated by the PhaseMap analysis based on the elemental details listed. Mole fraction of the elements in each phase is calculated to determine whether the experimental value matches with the empirical formula of the suggested compound. The elemental maps were verified with the data previously obtained on another SEM/EDS instrument (ASPEX Corporation, Delmont, PA).

RESULTS & DISCUSSION

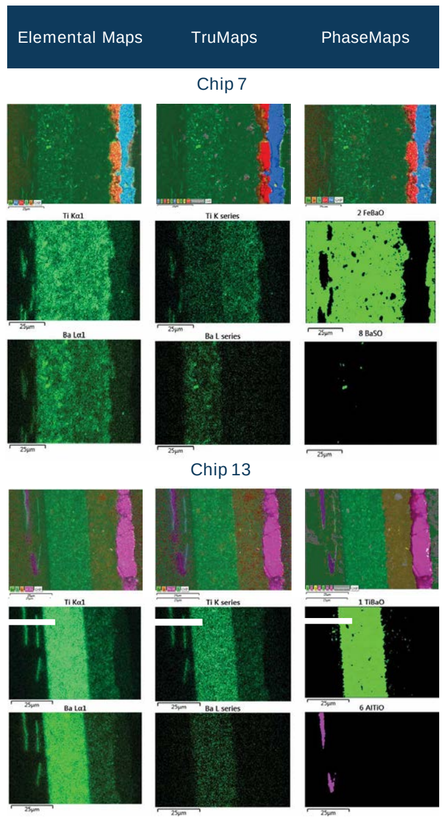

Figure 2. Unprocessed elemental maps, TruMaps, and PhaseMaps obtained from chip 7, J and 13. Top rows are composite images in each data mode.

- 10 out of 13 of the raw elemental maps were found identical to that previously obtained from the ASPEX SEM/EDS.
  - ►Interference between Ti and Ba were found in Chip 7, 13, and J.
- TruMap analysis helped to deconvolute the overlaying Ti and Ba layers.
  - ►Elemental maps should be processed with TruMap analysis if overlaying issue us suspected.
- PhaseMap analysis further confirmed that Ba was only present in primer layer in chip 7, 13, and J, but not in the E-coat.
- At this stage, mole fractions of the suggested chemical constituted in PhaseMap may not be conclusive to the true composition in each phase.
  - ►Deviation from the empirical ratios.
  - ►More data are needed to establish the acceptable range of experimental mole fractions from each of the inorganic compounds.

RESULTS & DISCUSSION (CONT'D)

Possible inorganic compounds	Expected X	Observed X
$BaSO_4$	Ba/S = 1.00	0.79 – 0.89
$H_2Mg_3(SiO_3)_4$(Talc)	Mg/Si = 0.75	0.61 – 0.78

Table 1. Mole fractions (X) calculated from the possible inorganic compounds obtained from PhaseMap analysis in the automobile paint systems.

CONCLUSION

- Similar elemental distributions were obtained in all 13 samples when compared to previously obtained data.
- TruMap analysis assisted to deconvolute overlaying principal X-ray lines in elemental mapping, e.g. Ti and Ba.
- PhaseMap analysis was also helpful in determining identifying possible inorganic compounds present in the automobile paint systems, e.g. $BaSO_4$.
- More data are needed for verifying the range of mole ratio of possible compounds present in each phase.
- The TruMap and PhaseMap analyses may be useful in providing investigative leads in forensic paint analysis for identifying and/or excluding suspected automobile samples.

REFERENCES

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