Geological Society of Malaysia Annual Geological Conference 2000
September 8–9 2000, Pulau Pinang, Malaysia

Recent EPMA Applications in Geology and Industry

G.H. TEH

Geology Department, University of Malaya
50603 Kuala Lumpur, Malaysia

Abstract

The electronprobe microanalyzer or EPMA has proved to be a useful analytical tool in earth science research and the industry in Malaysia. The EPMA has been harnessed in the characterisation and exploration for gold, nickel, cobalt, tin, and heavy minerals as well as mineralogical research where quantitative analyses and structural formulae are required. The majority of samples come from the industry where variations in the composition of samples and contaminants are important. Elemental distribution patterns revealed in X-ray maps are sought after in the types of remedial measures taken to improve the quality of the products.

Aplikasi Semasa EPMA dalam Geologi dan Industri

Abstrak

Penganalisamikro elektronprob atau EPMA terbukti penting sebagai peralatan analisis dalam kajian sains bumi dan industri di Malaysia. EPMA sesuai digunakan dalam pencirian dan ekplorasi emas, nikel, kobalt, timah dan mineral berat serta kajian mineralogi yang melibatkan analisis kuantitatif dan formula struktur. Kebanyakan sampel adalah dari industri dimana variasi kandungan sampel dan bahan pencemar adalah penting. Corak taburan unsur dalam peta sinar-X membantu penentuan jenis langkah baikpulih untuk mempertingkatkan kualiti hasilan.

INTRODUCTION

Since its commissioning in November 1997, the electronprobe microanalyzer or EPMA, at the Geology Department, University of Malaya, has proved to be a very useful analytical tool in earth science research and the industry in the country.

The majority of samples analysed involved simple qualitative or quantitative analyses of elements present. For samples that require detailed information on the distribution of the various concentrations of the elements present, the EPMA is very versatile in providing very informative X-ray maps.

MATERIALS AND METHODS

The EPMA is, in fact, a scanning electron microscope (SEM) with enhanced analytical capabilities. The EPMA available at the Geology Department, University of Malaya, is a highly-automated Cameca SX100 which is workstation-based, with full instrument control coupled with qualitative and quantitative software via windows and multi-task user environment. At the same time the EPMA utilises a fully integrated energy dispersive spectrometer (EDS) for fast, full spectrum scans of elemental compositions and 4 conventional wavelength dispersive spectrometers (WDS) with 12 analysing crystals for accurate compositional determinations of elements from Be to U.

Sample preparation sometimes require special techniques due to the varied shapes, sizes, hardness and value of the samples. For routine observation the samples are mounted on carbon tape or double-sided tape, while samples that require precise analytical data are normally mounted in resin and a good polished surface is required. The samples are then coated with carbon, gold or palladium for viewing with a secondary electron (SE) detector or a back-scattered electron (BSE) detector.

Geological samples that have been worked on include minerals, soils, clays, coals, ore minerals and rocks. Samples for mineral exploration for gold, cobalt, nickel, titanium and tin have successfully been determined by the EPMA (Teh, 1996a, 1996b; Teh *et al.*, 1997, 1998; Teh and Anisalimahwati bt Sulaiman, 1998). Meanwhile samples with geotechnical problems like marble at depth and burnt concrete have also been successfully solved by EPMA study.

Materials from the industry come in all sorts of compositions, shapes, sizes and types. Among the many items received are circuit breakers, filters, pipe coatings, paints, kaolinite, sludge, new material coatings, saw blades, corroded tubes, powders, air-filters, concrete, doorknobs, needles, contacts, precious stones, contaminated tubings, lenses, ceramics, stains etc. (Teh, 1999, 2000).

DISCUSSION AND CONCLUSIONS

On account of its ability to give precise analytical data on very small specimen size, the EPMA has found useful application in the exploration for gold in Malaysia (Figure 1). Characterisation and the geochemistry of the primary and alluvial gold grains have proved useful in the search for new gold deposits. Similar application has been applied for tin, nickel and cobalt exploration.

For mineralogical or petrographical research, there is a software on the SX100 where spot analyses can yield detailed data on the elemental composition (wt%), oxide composition (wt%), atomic composition, or the structural formula (according to Deer *et al.*, 1996) for individual minerals, inclusions or associated minerals (Table 1). Besides quantitative analyses and characterisation of minerals, ore minerals and heavy minerals, X-ray mapping has proved most useful in revealing the nature of various complex ores (Figure 2).

In the mineral industry, the EPMA has proved useful in locating the source of staining or discoloration of dimension stones, phosphorous contamination in TiO_2, as

Table 1: Quantitative analysis of diopside by EPMA with structural formula on the basis of 6 oxygens.

point n : 1 x= -10009 y= -6218 z= 8
diopside

elt.	peak pos.	peak (c/s)	backgr. (c/s)	i.x./i.std.	sig/k	beam	kv
						20.	20.0
Na	46401	30.35	8.55	0.01412	4.1		
Mg	38537	3791.13	95.43	0.14655	0.4		
Al	32469	145.11	28.70	0.00366	1.9		
Si	27733	14612.80	86.72	0.83994	0.3		
P	70383	2.75	2.10	0.00051	13.5		
K	42761	13.40	11.60	0.00066	6.1		
Ca	38388	4979.03	32.15	0.53130	0.4		
Ti	68277	2.05	2.30	0.00000	15.6		
Mn	24004	76.17	67.01	0.00081	2.6		
Fe	48081	29.15	5.85	0.00552	4.2		

Analysis no. 1 within pyro

element	k i.x./i.std.	k.ratio	wt. % concen.	normalized atom. c	compound	cocen.
Na :	0.0141	0.0005	0.139	0.13	Na2O	0.187
Mg :	0.1465	0.0599	10.879	9.66	MgO	18.041
Al :	0.0037	0.0014	0.248	0.20	Al2O3	0.469
Si :	0.8399	0.1812	25.803	19.82	SiO2	55.202
P :	0.0005	0.0001	0.013	0.01	P2O5	0.030
K :	0.0007	0.0001	0.008	0.00	K2O	0.009
Ca :	0.5313	0.1635	18.609	10.02	CaO	26.038
Ti :	0.0000	0.0000	0.000	0.00	TiO2	
Mn :	0.0008	0.0002	0.029	0.01	MnO	0.038
Fe :	0.0055	0.0036	0.440	0.17	Fe2O3	0.629
O			44.475	59.98 by stoic. with norm.		
total :			100.643			100.643

iteration : 5

pyroxene cations on 6. <o> basis

pyroxene			Wt.%	Cations
P2O5	0.0299	P	0.0131	0.0009
SiO2	55.2016	Si	25.8027	1.9773
TiO2	0.0000	Ti	0.0000	0.0000
Al2O3	0.4688	Al	0.2481	0.0198
Fe2O3	1.0585	Fe	0.7404	0.0285
MgO	18.0411	Mg	10.8793	0.9634
CaO	26.0381	Ca	18.6093	0.9993
MnO	0.0377	Mn	0.0292	0.0011
FeO	0.0000	Fe	0.0000	0.0000
Na2O	0.1874	Na	0.1390	0.0130
K2O	0.0092	K	0.0077	0.0004
total	101.0724			4.0039

wo: 50.89 en: 49.06 fs: 0.06

Ratio (Fe+Mn)/(Fe+Mn+Mg) = 0.12

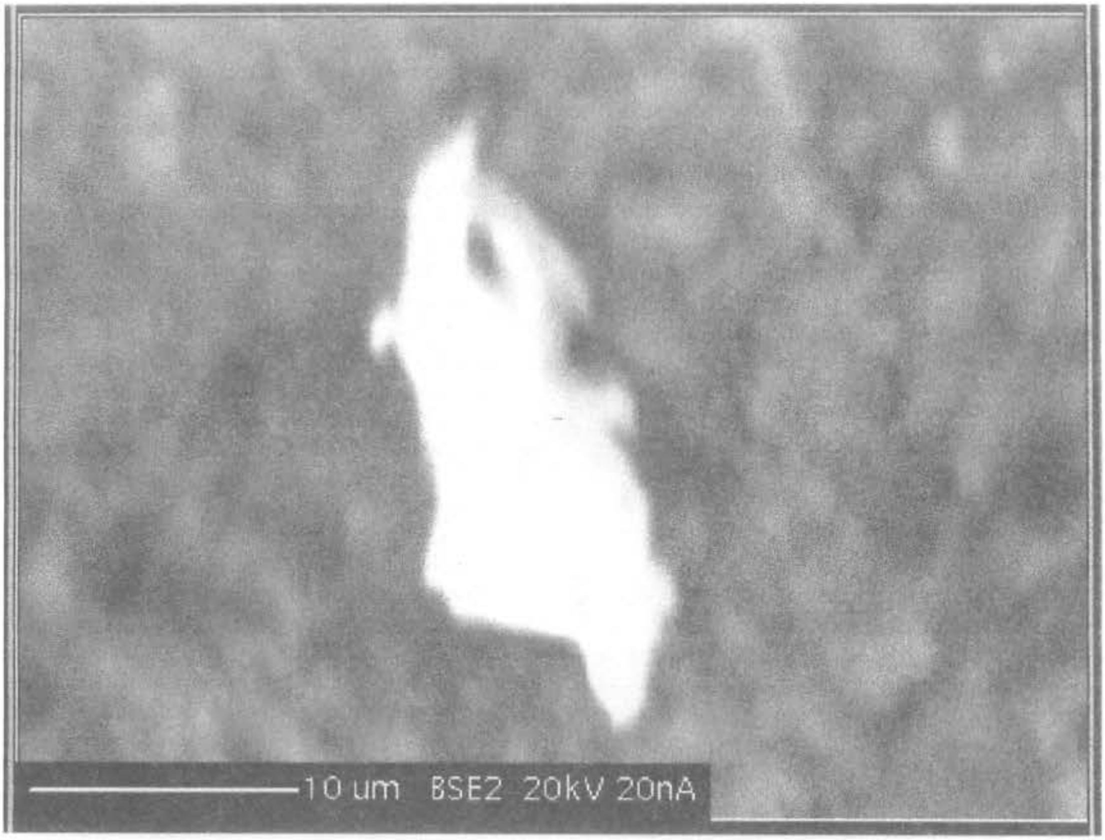

Figure 1: SEM photomicrograph of fine 'invisible' gold in fine grain rock made visible by the EPMA.

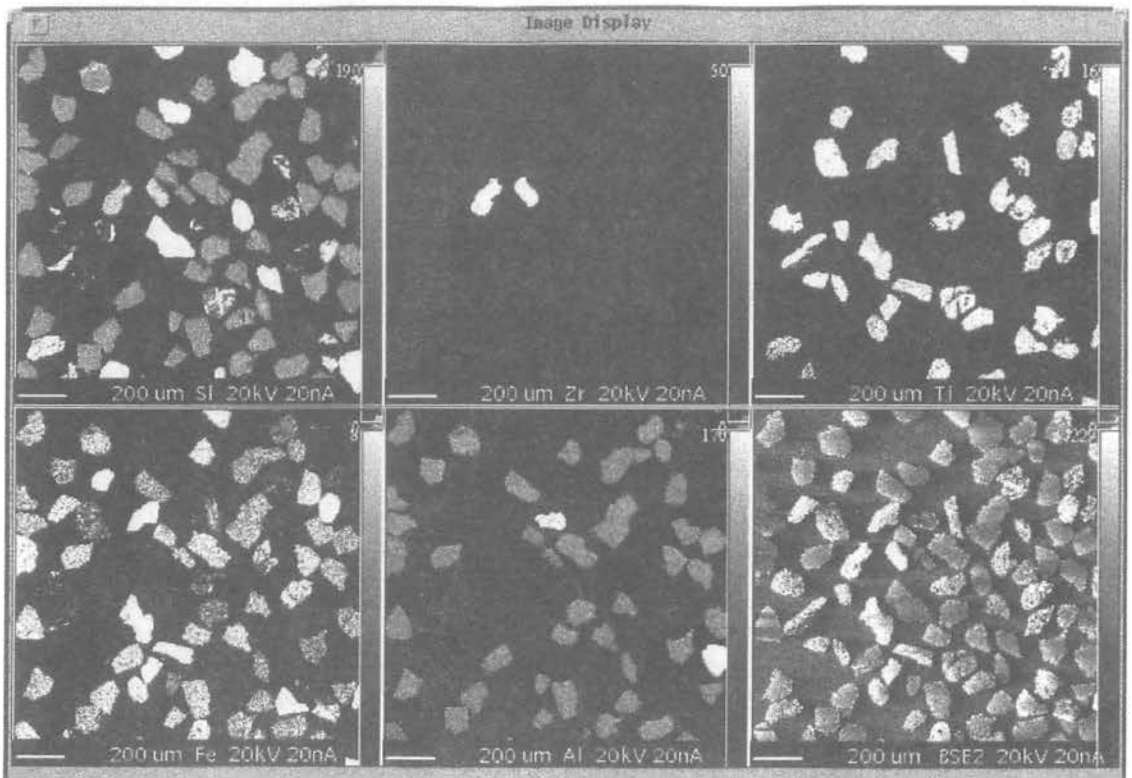

Figure 2: X-ray mapping used in heavy mineral exploration, a convenient way of separating quartz from ilmenite, zircon and tourmaline.

well as calc-silicate to mineral formation in marble (Figure 3).

In industry, where the SX100 gets most of its samples, the EPMA has proved to be a useful tool for the determination of variations in composition in samples and their contaminants, in particular their distribution patterns, which can be readily revealed by X-ray mapping (Figures 4–6). Armed with such information on elemental distribution from the EPMA, steps can then be taken to locate the sources responsible for such variations and remedial measures taken to improve the quality of the products.

ACKNOWLEDGEMENT

The permission granted by the various companies or organisations for the use of their data is greatly appreciated.

REFERENCES

Deer, W.A., Howie, R.A. and Zussman, J., 1996. An introduction to the Rock-Forming Minerals. 2nd ed. Longman, 696p.

Teh Guan Hoe, 1996a. Electronprobe X-ray microanalysis of geological samples. *In:* Proceedings 6th Scientific Conference, Electron Microscopy Society Malaysia, 26–27 October 1996, Petaling Jaya, p 26–27.

Teh Guan Hoe, 1996b. Electronprobe microanalyses and geochemistry of a complex Cu-Fe-Sn-Zn ore. *Warta Geologi*, Vol. 22, No. 6, p 389–394.

Teh, G.H., Helmi Mohd Latib and Zulpakar Mohamad Jusuh, 1997. Preliminary Electronprobe Microanalyzer (EPMA) characterisation of gold deposits of the Raub-Tersang-Selinsing-Penjom and Rusila areas. *Warta Geologi*, V. 23, No. 6, p 345–349.

Teh Guan Hoe, Helmi Mohd Latib and Zulpakar Mohamad Jusuh, 1998. Electronprobe microanalyzer (EPMA) characterisation of Malaysian gold deposits. *In:* Proceedings 7th Scientific Conference of the Electron Microscopy Society Malaysia, 27–28 November 1998, Ipoh, Malaysia, p 78–82.

Teh, G.H. and Anisalimahwati bt Sulaiman, 1998. Preliminary geochemistry and characterisation of alluvial gold from the Jeli and Sokor areas, Kelantan. *Warta Geologi*, V. 24 No. 6, p 297–301.

Teh, G.H., 1999. Applications of EPMA (Electronprobe Microanalyzer) in industry. *In:* Proceedings 8th Scientific Conference of the Electron Microscopy Society Malaysia, 2–4 December 1999, Genting Highlands, Pahang, Malaysia, p 60–62.

Teh Guan Hoe, 2000. Electronprobe Microanalyzer (EPMA) application in geology and the industry in Malaysia. *In:* Proceedings, 7th Asia-Pacific Electron Microscopy Conference (7APEM), 26–30 June 2000, Singapore, p 296–297.

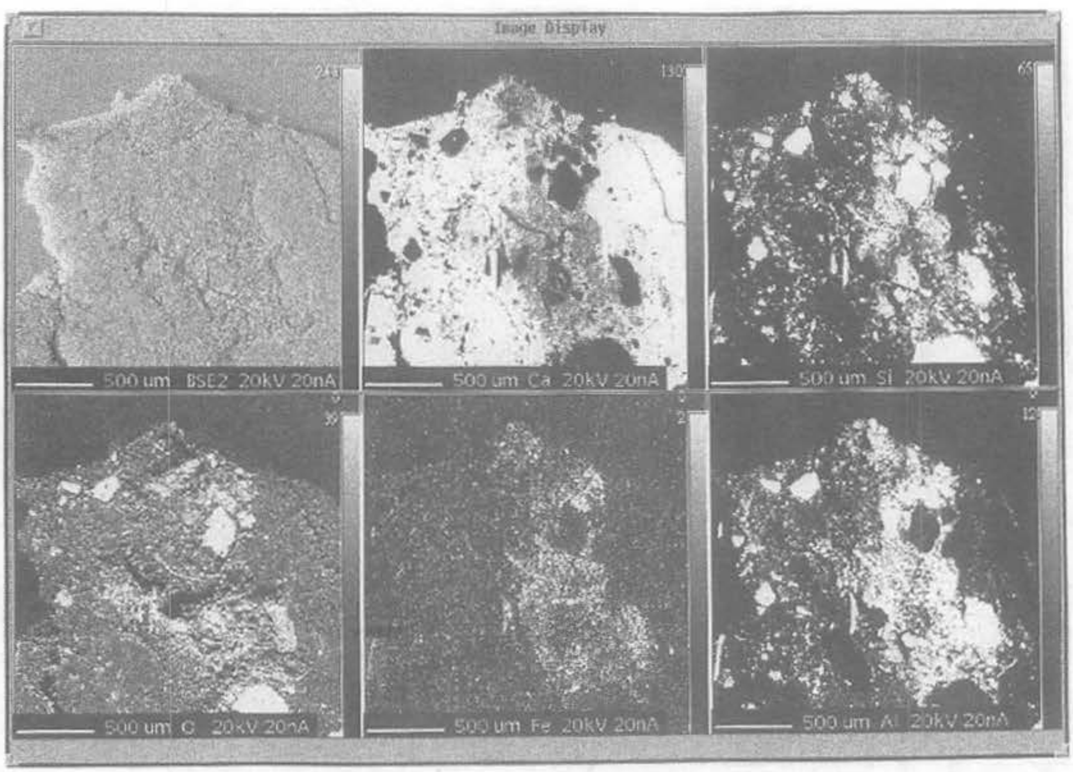

Figure 3: Marble at depth weakened by formation of pockets of calc-silicate minerals.

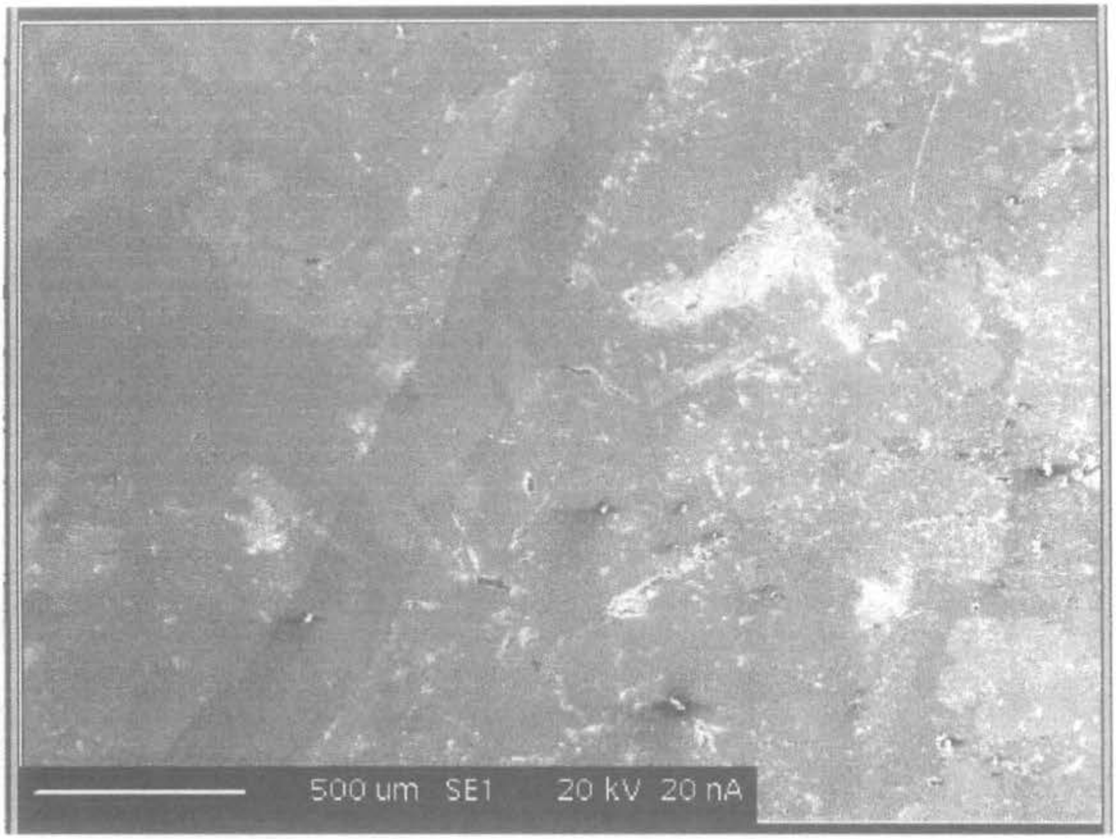

Figure 4: SEM photomicrograph of discoloration of surface of dimension stone due to development of micro-channels penetrated by undesirable solutions (left – normal, right – decolored).

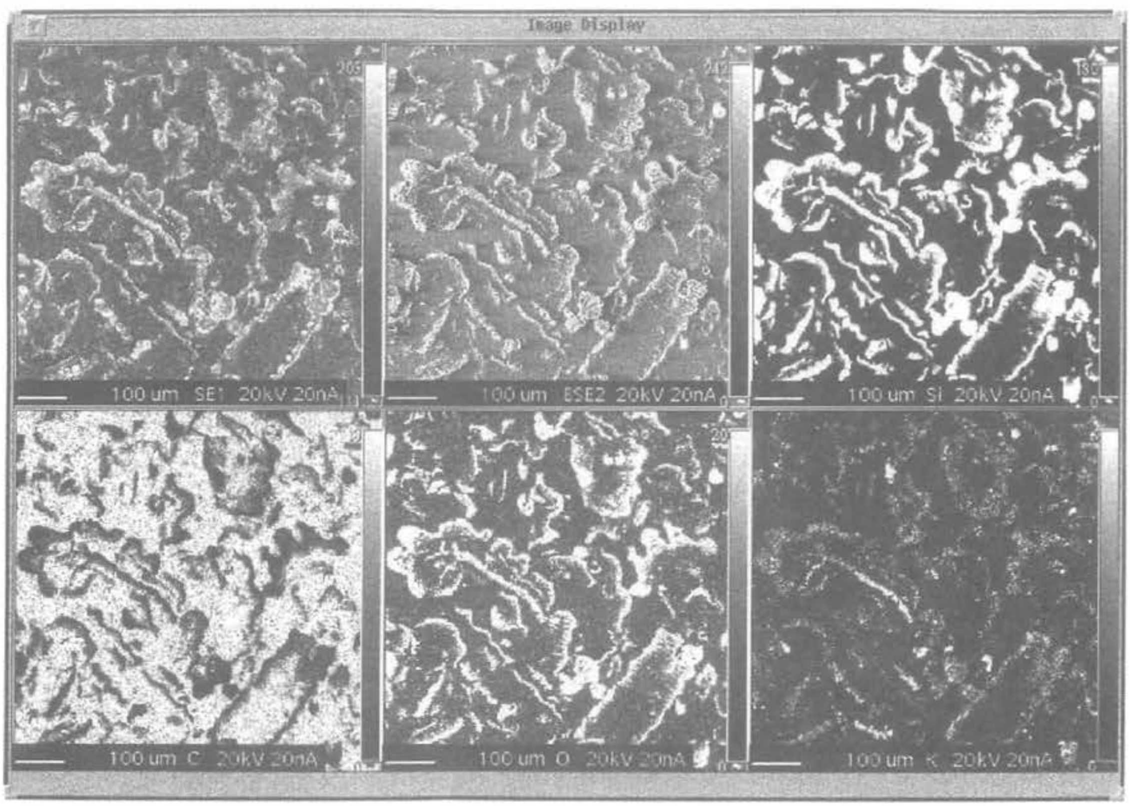

Figure 5: X-ray map of burnt rice husk, with abundant cryptocrystalline quartz, used in the filter industry.

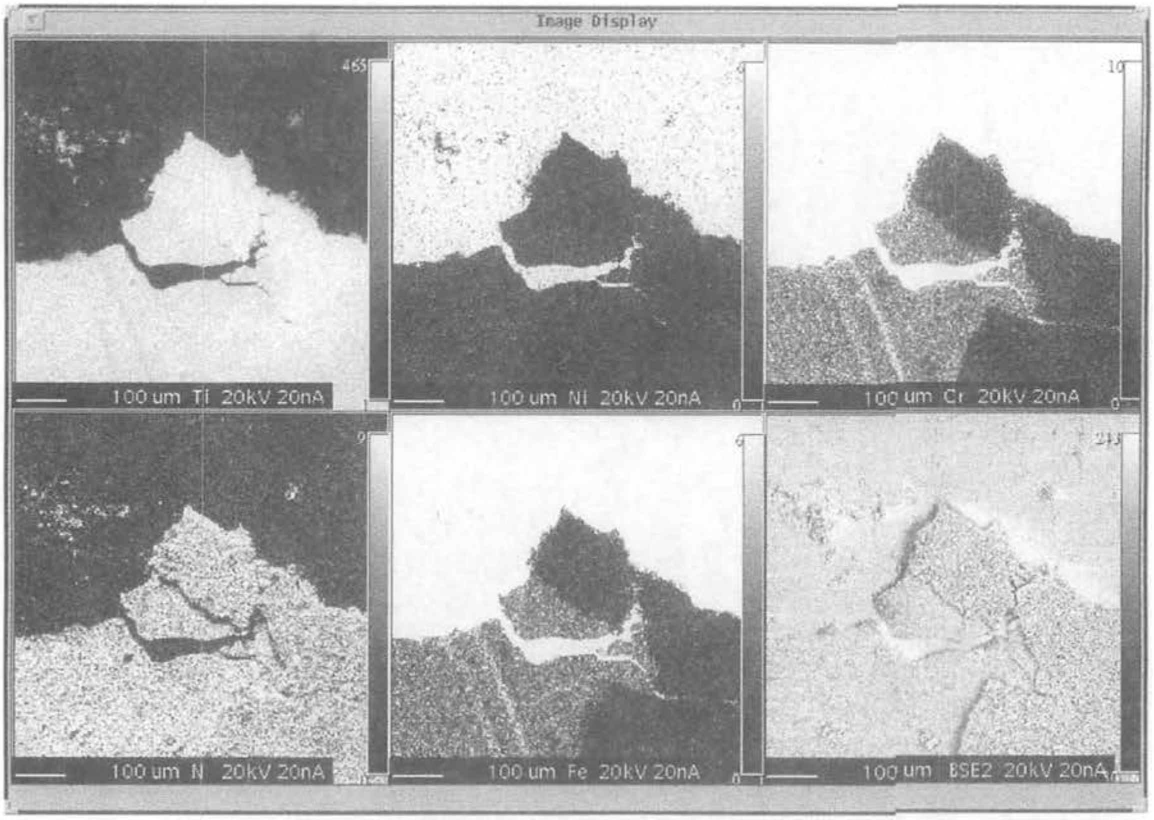

Figure 6: Plasma coating of steel plate with gold-coloured TiN.