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PROCEDURES FOR ANALYSIS OF DEBRIS RELATIVE TO SPACE SHUTTLE SYSTEMS

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SUMMARY

The Microchemical Analysis Branch at Kennedy Space Center is requested to provide elemental and phase analysis of debris collected from facilities, miscellaneous components and materials, the Space Shuttle and its systems and Space Shuttle and expendable launch vehicle payloads. Debris are collected on cloths, swabs, glove fingers, transparent sticky tape, razor blades, filters and large sampling containers.

A combination of optical microscopy (OM), scanning electron microscopy (SEM) with energy dispersive spectrometry (EDS), analytical electron microscopy (AEM) with wavelength dispersive spectrometry (WDS) and X-ray diffraction (XRD) is well suited to the investigation of debris.

Dry bulk particulates can be examined with a stereobinocular microscope to give a good idea of their characteristics, such as dryness, homogeneity, uniformity and particle size. If debris are too large for examination by transmitted light OM, the particles should be crushed until their thickness is reduced to about 30 micrometers. Particle sizes are measured using an optical micrometer or SEM image, and the linear extension values are used. The linear extension values represent the actual diameter of regular particles and the projected or statistical diameter of irregular particles.

The estimated volume percent of each component or particle type represents the relative area occupied by it on a particular planar surface. Estimation of volume percent for each component is made by comparing the observed distribution with a standard visual estimation chart. The error from this technique is about 5 percent as determined by comparison of the estimated volume percent of each particle type with the actual volume percent obtained by the point count method.

The important optical properties for OM examination are transparency, opacity, color, refractive index, birefringence, size, crystalline or noncrystalline properties and shape and morphology of crystallites. These characteristics yield primary criteria for debris identification, because certain species often have rather specific optical properties. Particles such as fine grained materials, opaque inclusions, poorly crystallized materials, cryptocrystalline materials and needle-shaped crystals which cannot be separated from the debris for refractive index determination are not readily identified by OM.

The SEM has unique capabilities for analyzing surface morphology. It uses electrons for image formation. Electrons have a much shorter wavelength than light photons, making them capable of generating images with high resolution, high depth of field, and three-dimensional appearance.

For SEM analysis, particles previously classified on the basis of morphology and color by OM are picked out individually. Small classified components are mounted directly on a highly polished beryllium surface or polished carbon planchet or fastened to a carbon planchet by double stick carbon tape or to an aluminum block by double stick clear tape. Particles smaller than 3 micrometers are embedded in a film of collodion, and the film is removed, floated in water and transferred to the SEM stub. The collodion is then dissolved in acetone, and the particles remain on the stub. Debris can also be coated with carbon or gold to allow better conductivity in the SEM.

For most EDS analyses, however, the debris are usually not coated with carbon or gold to avoid any X-ray peak overlapping problems with unknown samples. The energy characteristics of X-rays generated in the sample are used to determine the elemental composition of the region of debris being bombarded by electrons. Boron (B) and hydrogen (H) can not be detected by EDS. The elemental composition of most debris is listed in order of decreasing abundance. The order is based upon peak intensities recorded during analysis, where the peak intensity is roughly proportional to the element concentration. The elemental data can be used as an aid for later phase identification by XRD.

The AEM is a microanalytical tool combining SEM and WDS. Samples as small as 1 cubic micrometer can be analyzed by AEM, and detection limits are as low as 0.001 weight percent for nearly all elements. Sample preparation methods for AEM are similar to those for qualitative analysis by SEM. For best quantitative analysis results, the specimen should be unetched and polished so that the surface is as flat and scratch-free as possible. The debris must be impregnated or potted with resin, then sectioned through after the resin has hardened. The sectioned surface is polished through the steps of rough grinding, fine grinding, rough polishing and final polishing.

For WDS analysis, debris are usually polished and coated with carbon. Qualitative analysis by WDS is based on the ability of a spectrometer system to measure characteristic X-ray line energies and relate them to the presence of specific elements. The characteristic X-ray spectra constitute "fingerprints" for identification of the elements. WDS's good resolution and peak-to-background ratio and its ability to show more members of the family of X-ray lines for a given element allow positive detection and identification of elements at low concentrations. Quantitative analysis by WDS involves measuring the intensity of spectral lines, calculating intensity values and converting them into chemical concentrations using various correction methods.

XRD techniques can be applied only to crystalline materials. The crystal planes of each crystalline material form a unique X-ray pattern or signature. Qualitative phase identification by XRD is determined by the position of two theta(d-spacing) and the intensity of the X-rays. Typical XRD data are read as two theta versus intensity. The Microchemical Analysis Laboratory maintains an accuracy of one part per thousand in the position of two theta. With this accuracy of alignment, most crystalline compounds, including solid solution series, can be identified.

PROCEDURES FOR ANALYSIS OF DEBRIS

I-INTRODUCTION

1.1 BACKGROUND:

Debris samples collected from various Space Shuttle systems have been submitted to the Microchemical Analysis Branch during the last several years. These samples were mainly from the External Tanks, hydrazine systems, chloroflourocarbon (CFC) replacement projects, payloads, GH_2 and GOX lines, Payload Changeout Rooms, LH_2 systems, LOX lines, metal bolts, Mobile Launcher Platforms, Orbiter Processing Facilities, Orbiter Thermal Protection Systems, paints, Solid Rocket Boosters, Vertical Processing Facility, weld metals, ground support equipment and natural environments.

1.2 OBJECTIVE:

The need for debris analysis is likely to continue in the future. Therefore, this investigation was initiated to develop optimal techniques for the analysis of contaminants. These include analysis of debris using optical microscopy, scanning electron microscopy with energy dispersive spectrometry, analytical electron microscopy with wavelength dispersive spectrometry and X-ray diffraction.

II-TECHNICAL APPROACH

A combination of optical microscopy (OM), scanning electron microscopy (SEM) with energy dispersive spectrometry (EDS), analytical electron microscopy (AEM) with wavelength dispersive spectrometry (WDS) and X-ray diffraction (XRD) has been used to characterize debris.

OM provides information on the morphology and size of crystallites, particle sizes, amorphous phases, glass phases, poorly crystallized materials and estimated volume percentage of each component. SEM with EDS provides information on the surface morphology and qualitative elemental content of debris and is used to aid XRD phase analysis. AEM with WDS provides information on the detailed surface morphology and quantitative elemental content of debris.

Occasionally it is not possible to identify the debris using any single technique. In these cases, a combination of instruments is used to positively identify debris.

III-SAMPLE DESCRIPTION

3.1 Debris Origins:

Table 1 lists sample origination points and the number of projects received from each point during the last six years. The major origination points of the samples were facilities, miscellaneous components and materials, payloads (Shuttle and Expendable Launch Vehicle) and the Shuttle and its systems.

Table 1. Debris Origination Points and Number of Related Projects

SAMPLE ORIGINATION POINT	**NUMBER OF PROJECTS**
Facilities	
CCAFS	
a. Complex 17	1
b. Complex 34	3
c. hangars	5
d. Vertical Integration Building (VIB)	1
ESA-60 (fuel and oxidizer scrubbers)	2
Hypergol Maintenance Facility (HMF)	2
K6-1200D	1
Launch Complex 39 (LC-39) A&B	
a. general	32
b. Payload Changeout Rooms (PCR)	6
MRC storage crib	1
NASA Shuttle Logistics Depot (NSLD)	2
Operations & Checkout Building (O&C)	8
Orbiter Processing Facility (OPF)	
a. general	14
b. bridge/bucket hoists	5
Payload Hazardous Servicing Facility (PHSF)	3
Shuttle Landing Facility (SLF)	2
Spacecraft Assembly & Encapsulation Facility (SAEF-2)	1
Vandenberg AFB breathing air system	1
Vertical Processing Facility (VPF)	4
Misc. Components, Materials, etc.	
aluminum tubing	1
antennae door	1
battery plates & casing	3
CFC replacement project	3
concrete samples	3
DMES waterproofing compound	2
electrical connector contact/terminal assy.	7
grease, oil, lubricant	17
hat fuse pin	1
hydraulic fluid	7
Hydra-Sets	1
Hypalon topcoat, Elephant Hide coating	2
magnetic computer tape	1
metal bolts	3
paint samples	5
printed circuit board	1
solder	4
TVL tubing	1
weld metal	2

Table 1, continued

Payloads (Shuttle and Expendable Launch Vehicle)

Astro 1	5
Atlas	4
Gamma Ray Observatory (GRO)	1
Lageos/IRIS spacecraft	1
Magellan	1
Spacelab D2	1
Super-Fluid Helium On-Orbit Transfer (SHOOT)	1
Tethered Satellite System (TSS)	2
Ulysses	4
US Microgravity Payload (USMP)	4

Shuttle

Auxiliary Power Units (APU)	7
External Tanks (ET)	
a. general	11
b. liquid hydrogen (LH_2) Lines	23
c. liquid oxygen (LO_2) Lines	9
d. pneumatics	6
Mobile Launcher Platforms (MLP)	188
Orbiter	
a. general	37
b. Main Propulsion System (MPS)	15
c. Maneuvering/Reaction Control Sys. (OMS/RCS)	14
d. particles from post-landing wipes & scrapes	24
e. payload bay doors	4
f. Thermal Protection System (TPS)	22
OV-102	14
OV-103	27
OV-104	11
OV-105	7
pyrotechnics	3
Solid Rocket Boosters (SRB)	17
Space Shuttle Main Engines (SSME)	8

Systems

gaseous helium (GHe)	9
gaseous nitrogen (GN_2)	5
gaseous oxygen (GO_2)	13
Hypergol Vapor Detection System (HVDS)	1
liquid hydrogen (LH_2)	7
LH_2, service GHe	16
LH_2, service GN_2	3
liquid oxygen (LO_2)	19
N_2H_4 (hydrazine)	6
N_2O_4 (nitrogen tetroxide)	1
Power Reactant Storage and Distribution (PRSD)	2
Purge, Vent and Drain (PVD)	5
Spacecraft Processing	5

3.2 Debris Sampling:

The wide variety of debris sampling procedures can be conveniently considered under two headings: unfractionating and fractionating. Unfractionating sampling procedures involve the collection of a single gross sample, while fractionating procedures involve the collection of a number of separate fractions of the gross sample. Table 2 categorizes several sampling methods as unfractionating and fractionating (1). This study covers only those techniques applicable to the sampling procedures used at the Kennedy Space Center (KSC).

Table 2. Categorization of Unfractionating and Fractionating Sample Collection Methods

UNFRACTIONATING METHODS	**FRACTIONATING METHODS**	
	During Initial Collection	**On Collected Gross Sample**
tape	electrostatic precipitator	particle picking
snow surface	thermal precipitator	magnetic separation
foliage	impactor	density gradient
dust fall jar, cloth or paper	centrifugal separation	elutriation
impinger	cyclone	sedimentation
cyclone		low temperature ashing (LTA)
filter		solvent extraction
		sublimation
		sieve

3.2.1 Debris Collected on Cloths:

This method is a very common procedure for collecting the gross particles from any surface. The debris are usually collected on either lint-free cloth (Figure 1) or Texwipe (Figure 2). The loose particles adhere to the cloth. The particles are protected by folding the cloth and can be removed by scraping the cloth with a sharp needle. The side-to-side motion of the needle against the cloth produces vibration that frees the particles, allowing them to fall onto white paper placed beneath the cloth. The particles are then transferred into a clear plastic petri dish for further study with OM, SEM with EDS and AEM with WDS.

3.2.2 Debris Collected on Swabs and Glove Fingers:

These methods are used in cases where the amount of particles in the samples is small and/or the sample origination area has accessibility limitations. Particles can be collected by smearing swabs (Figure 3) or glove fingers (Figure 4) on the sampling surface. Usually only a small amount of particles can be collected on each swab or glove finger, so several samples are necessary to collect enough debris for analysis. The samples are protected by wrapping the swabs or gloves in plastic. The particles are removed from the swabs or glove fingers by gentle scraping with a needle and allowing the particles to fall onto a clean glass slide.

3.2.3 Debris Collected on Sticky Tape and Razor Blades:

Two quick means of collecting samples are transparent sticky tape (Figures 5 and 6) and razor blades (Figure 7).

When sticky tape is applied to the sampling surface and removed, loose particles adhere to the tape. The tape is then placed on a clean cellophane sheet or a glass slide to protect the sample. Sticky tape collection, however, can interfere with organic sample analysis due to organics in the adhesive. This method is also the most difficult for the analytical microscopist when trying to remove the particles from the adhesive. Particles are removed from the tape for microscopic examination by picking with a needle. If removal of all particles from the tape is necessary, it is best to use a tape that is soluble in some organic solvent.

For particles that are harder to remove, a razor blade can be used to scrape them from the sampling surface. The particles are then transferred to a petri dish or glass slide for microscopic examination.

3.2.4 Debris Collected on Filters:

Filters are the most useful devices for separating suspended particles. Most filters are made of paper, but filters are available in almost every conceivable fiber, from natural sources, such as wood pulp or cotton, to synthetics, such as nylon, Teflon, glass or metal (Figure 8).

3.2.4.1 Fiber Filters:

Fiber filters have an extremely high collection efficiency. However, smaller particles tend to penetrate more deeply and become lodged in the tangle of fibers. In attempting to remove small particles, contamination of the debris with fiber fragments is very likely. Gentle tapping of the filter is probably the best way to free at least large particles from the fibers. Fiber filters are not generally recommended for collecting samples with small amounts of debris.

3.2.4.2 Membrane Filters:

Membrane filters are porous polymeric films manufactured from polymers which may be resistant to heat, acids, bases and/or organic solvents. They allow low penetration of fine particles and have good solubility and good optical properties. The surfaces of membrane filters are smooth, and particles can be removed for analysis with relative ease. Also, a portion of the filter can be cut out, solvent-vapor treated to grip the particles, and gold or carbon coated for direct OM and SEM observation. If the selected filter material is soluble in some organic solvent, all of the non-soluble particles can be isolated for examination.

3.2.4.3 Nuclepore Filters:

Nuclepore filters have pores that are uniform in diameter and nearly cylindrical in shape. The surfaces of these filters have an almost complete absence of rough texture. Particles can be easily examined on the surface of the Nuclepore filter in any refractive index medium, but can just as easily be removed.

3.2.5 Debris Collected on Other Media:

Samples having large volumes of particles can be collected with almost any kind of sampling device. Figures 9, 10, 11 and 12 show debris collected on an aluminum dish and clear plastic petri dishes. When collecting samples in dishes, great care must be exercised to make certain the samples are not contaminated during sampling and before analysis.

IV-SAMPLE PREPARATION

4.1 Optical Microscopy:

When particles collected by one of the methods discussed in section 3.2 arrive at the lab, they are first examined optically. NASA's Microchemical Analysis Branch has prepared a special base for examination of particles with a stereobinocular microscope (Figures 13 and 14). One half of the base is white, for examination of dark colored particles, and the other half is black, for examination of light colored particles. Figure 15 is an optical photomacrograph of particles using the black half of the base as the background. Figure 16 is an optical photomacrograph of the same particles in front of the white half of the base.

With the aid of the black and white base, the particles are classified into components according to color and texture, and the visual volume percent of each component is estimated. The particles are then prepared for examination by SEM and AEM (Table 3).

Table 3. Example of Component Classification with Estimated Volume Percentages and EDS Results

Component ID	Elemental Analysis by EDS		Part. Size um
	Major	Minor	
Sample 1			
Metallics (T)	Al		1-50
Black material (T)	Fe, Si, Cl, K	Cr, Al, S	1-70
Organics (100)	Teflon		1-500
Sample 2			
Metallics (T)	Fe, Cr, Al, Cu	Ni, Sn	1-80
Black material (T)	Fe, Cr, Si, Cl	Al, S	1-110
Organics (100)	Teflon		1-600

(100): estimated volume percent
T: trace amount
um: micrometers

4.1.1 Bulk Samples:

Dry bulk particulates collected on cloths, swabs or glove fingers can be examined with a stereobinocular microscope to gain an understanding of their nature, e.g. dryness, homogeneity, uniformity, particle size, etc. A representative sample is then removed with a microspatula and transferred to a microscope slide. A drop of a volatile mounting medium is then placed over the sample. The sample is dispersed uniformly by sliding a cover slip over the drop with a circular motion. If the particles are too large for transmitted light optical examination, they should be crushed using an agate mortar and pestle. The optimum particle size for polarized transmitted light optical analysis is about 30 micrometers in thickness.

4.1.2 Particles on Sticky Tape:

Two methods for removing particles from sticky tape were mentioned in Section 3.2.3, picking with a needle and dissolving the tape's adhesive. A needle can be used if examination of only a small number of particles is necessary. If removal of all the particles is necessary, solvent action is used to dissolve the tape's adhesive. First, the gummed surface of the tape is exposed, and a 2 inch strip is put in a centrifuge tube with xylene. When the tape has completely dissolved, the particles are centrifuged to the bottom of the tube. The particles at the bottom of the tube are then transferred to a glass slide and dried before further analysis.

For quick examination, the tape sample is placed sticky side down on a glass slide and observed directly. The particles can be removed from the tape by introducing benzene between the slide and the tape and stripping off the tape, leaving the adhesive and embedded particles on the slide.

4.1.3 Particles on Filters:

Debris are most commonly received at KSC on paper, cellulose fiber, glass fiber or membrane filters. Particles collected on filters should first be studied directly in reflected light. Transmitted light may also be helpful in locating embedded particles. If only a few particles are present, they should be removed with a tungsten needle and transferred to a clean glass slide for further analysis. If there is a large number of particles on the filter, the particles can be viewed in transmitted light by immersing a portion of the filter in a refractive index oil. The index number of the oil should be close to that of the filter - about 1.54 for paper filters, 1.52 for glass fiber filters and 1.51 for membrane filters. The filter essentially disappears in the proper medium, and the particles lie nearly in a single optical plane. However, it is difficult to isolate individual particles from the oil for further analysis.

If removal of all the particles from a filter is necessary, different procedures are followed for each type of filter. For paper or cellulose fiber filters, low temperature ashing is used to eliminate the filter and the organic portion of the sample, leaving only the inorganic particles. Glass fiber filters should generally be avoided, since small particles can become trapped between the fibers, however a majority of the particles can be removed ultrasonically. Particles can be removed from soluble membrane filters by dissolving all or a portion of the membrane in a centrifuge tube of solvent, usually acetone. The particles are then centrifuged to the bottom of the tube, and the suspended particles are dried by evaporation.

4.1.4 Special Sampling Procedures:

Many particle contamination problems brought into the laboratory are unique. The actual contaminated hardware or component may be sent to the lab, leaving removal of the particles up to the investigator. Single, few or many particles may be present on or embedded in hard, soft or hard-to-reach surfaces. Such particles may be removed using tools such as needles or tweezers. Flushing with alcohol may also be necessary. A thorough OM examination of the contaminated surface allows the investigator to choose an appropriate handling procedure.

4.2 Scanning Electron Microscopy with Energy Dispersive Spectrometry

SEM and EDS analysis for qualitative elemental identification require samples to be mounted in some way on an electrically conductive stub. The method used for mounting a sample depends on the size and number of the particles.

4.2.1 Bulk Particles:

Dry bulk samples can be mounted on double-stick carbon tape applied directly to the stub. The particles are transferred to the tape either by spatula or by pouring them onto the tape. Loose particles are shaken from the tape to avoid charging effects in the SEM. Particles can also be attached to the SEM stub with carbon paint or a polymer coating, such as collodion. A thin layer of the mounting medium is applied to the stub, and the particles are transferred to the stub just as the medium begins to dry.

4.2.2 Individual Particles:

Particles up to 3 micrometers can be mounted using the same methods used for mounting dry bulk samples. They can also be mounted in liquid suspension on a glass cover slip attached to the SEM stub. Another mounting method involves first embedding the particles in a film of collodion. The film containing the particles is then floated in water and transferred to an SEM stub. The collodion is dissolved in acetone, and the particles remain on the stub.

Particles larger than 3 micrometers can be picked out and classified on the basis of morphology and color. The classified particles are then mounted onto a surface that will not contribute a detectable characteristic X-ray signal of its own, such as highly polished beryllium (Figure 17), polished carbon planchet (Figure 18), double-stick carbon tape on a carbon planchet (Figure 19) and/or double-stick clear tape on an aluminum block (Figure 20). Figure 21 is a high magnification OM photomacrograph of classified particles mounted on double-stick carbon tape. A thin hydrocarbon oil can be used to help particles stick to a beryllium block. If carbon is to be accurately detected in the sample, however, hydrocarbon oil can not be used.

4.3 Analytical Electron Microscopy with Wavelength Dispersive Spectrometry:

For qualitative elemental identification using AEM and WDS, the same sample preparation methods that are used for SEM and EDS analysis can be used. However, quantitative analysis requires special sample preparation methods. For quantitative analysis, the sample should be as flat and scratch-free as possible. The sample should not be etched, so its topography and surface chemistry remain unaltered. Table 4, found at the end of section 4.3, lists the procedures for polished and thin sample preparation for quantitative analysis.

4.3.1 Mounting Samples in Resin:

Preparing a sample suitable for quantitative analysis is accomplished by impregnating or potting the debris with resin and polishing the sample to obtain a smooth surface. Cured resins are safe and convenient for supporting and orienting samples. They also protect critical and/or delicate portions of the sample. The type of resin used to prepare the sample depends on the characteristics of the sample.

4.3.1.1 Spur Resin:

Spur resin is used for the impregnation of weak, porous and friable particles. It has an extremely low viscosity and vapor pressure, allowing it to readily penetrate into sample interiors. The refractive index of cured spur resin is 1.506. Spur resin is prepared using the listed amounts of the following:

Ingredient	Weight in Grams
4-vinyl cyclohexene dioxide (VCD)	10
diglycidyl ether of prolypropylene glycol (DEP)	4
nonenyl succinic anhydride (NSA)	26
dimethyllaminoethanol (DMAE)	0.4

After mixing the ingredients thoroughly, the resin is left at room temperature for 2 or 3 minutes (until clear) before it is used to impregnate samples. The shelf life of the uncured resin is limited to two weeks. Using a combination of room temperature curing and oven curing, the curing time of spur resin is in excess of two days.

4.3.1.2 Epo-Tek Resin:

Epo-Tek resin is used primarily for mounting polished faces onto glass slides before thin-sectioning, but it can also be used to cement cover glasses over thin specimens. Epo-Tek is optically clear and has a very high bonding strength. An additional advantage of Epo-Tek is that it does not form bubbles during curing. Epo-Tek is prepared using 3 parts by volume of Epo-Tek 301, part A and 1 part by volume of Epo-Tek 301, part B (hardener). Epo-Tek resin must be used immediately after mixing, because it gels after only 10 minutes. Epo-Tek is cured at room temperature for about 16 hours. The refractive index of cured Epo-Tek is 1.530.

4.3.1.3 Epon 815 Resin:

Epon 815 resin is used for potting debris samples. It is ideal for use with non-friable debris. Epon 815 resin is prepared using 89 weight percent Epon 815 and 11 weight percent TET (hardener). Like Epo-Tek, Epon 815 must be used within 10 minutes after mixing. The curing time of Epon 815 is at least 8 hours at room temperature. The refractive index of cured Epon 815 is 1.574.

4.3.2 Sectioning Mounted Samples:

After the debris are impregnated with resin and the resin has cured, the debris and the resin are sectioned using a high speed precision saw. It may be necessary to resection a sample to insure thorough penetration of the impregnant into the next layer of interest. Reimpregnation is required when unimpregnated spots show even after a sample is resectioned.

4.3.3 Grinding:

4.3.3.1 Rough Grinding:

Rough grinding removes surface damage incurred during sectioning and produces a surface that is as flat as possible and free of pits, scratches, saw marks and intergranular relief. The types and sizes of abrasives, wheel speeds and wheel coverings are all critical factors in rough grinding and are determined by the characteristics of the sample material (2).

4.3.3.2 Fine Grinding:

Fine grinding minimizes or eliminates the surface damage caused by rough grinding. Fine grinding should be performed until the rough surface of the section is reduced to approximately 50 to 100 micrometers. Grinding with silicone carbide paper for 3 to 15 minutes is generally preferred for hard to very hard materials.

4.3.4 Polishing:

4.3.4.1 Rough Polishing:

Rough polishing is required to remove damage caused by fine grinding and to prevent edge rounding and pitting during final polishing. A sample properly prepared for final polishing must have adequate and planar material removal, and this is achieved by using diamond paste suspensions during rough polishing. Diamond paste sizes used for rough polishing range from 9 to 6 microns, and a lapping time of 1 to 3 minutes is required.

4.3.4.2 Final Polishing:

Cerium-chromium (Ce-Cr) oxide and diamond abrasives are most commonly used for final polishing. Aluminum oxide and cerium oxide have also been tried with varying success. Ce-Cr abrasive is 0.05 micrometers in size, and a 1 to 3 minute polishing time produces the best results for final polishing of most materials. A fine (1 or 0.25) diamond abrasive should be used for final polishing of hard to very hard materials. Figures 22, 23, 24 and 25 show mounted and polished samples of debris, metal particles, standards and concrete.

Table 4. Procedures for Polished and Thin Section Sample Preparation

Procedure	Polished or Thin Section (P or T)	Abrasives, Resins or Saws	Size of Abrasive	Lapping Time (min.)	Wheel Cover Type
Sectioning	P, T	high speed tub saw or Isomet low speed saw	-	-	-
Impregnation, Potting	P, T	Spur or Epon	-	-	-
Mounting	T	Epo-tek	-	-	-
Resectioning	P, T	Isomet low speed saw	-	-	-
Rough Grinding	P, T	SiC	60 grit-400 grit	3-15	SiC pap. Texmet
Fine Grinding	P, T	SiC diamond	600 grit - 15 um	3-15 1-5	SiC pap. Texmet
Rough Polishing	P, T	diamond	9 um or 6 um	1-3 1-3	Texmet Texmet
Fine Polishing	P, T	diamond Ce-Cr	3-.25 um .05 um	1-3 1-3	Texmet Gold Micro-Supreme

4.4 X-Ray Diffraction:

A particle size of 30 micrometers is ideal for XRD work. Larger particles cause the X-ray film to be spotty, and particles ground to smaller sizes may have a damaged crystal structure.

4.4.1 Sample Preparation for the Powder Camera:

4.4.1.1 Glass Fiber Support:

A glass fiber between 3 and 5 micrometers in diameter provides a good support for particles. Particles that have been ground to about 30 micrometers can be mounted on the end of the glass fiber using a paraffin wax binder (Figure 26). This mounting technique is useful for analyzing very small amounts of particles and materials that are poorly crystallized. One drawback to using paraffin wax as a binder is that the X-ray reflections from the wax could be overlapped with those of the particles.

4.4.1.2 Glass Capillary Tube:

X-ray reflections from the mounting medium can be prevented by using a glass capillary tube. However, this mounting technique requires a large amount of particles to produce a readable pattern. Capillary tubes with an internal diameter of 0.3 or 0.5 millimeters can both be used. While the larger diameter tubes are easier to load, the smaller ones are preferable for more accurate work.

The key to loading particles into a capillary tube is introducing only a small amount of particles at a time. Tapping the open end with a spatula blade or rubbing it gently with a nail file helps the particles to move slowly and evenly down to the sealed end. Trapped air bubbles can be expelled by gently tapping the side of the tube. This process should be repeated until a solid filling of particles occupies 1 to 2 centimeters of the sealed end of the tube. The open end of the tube is then sealed using a microflame (Figure 27).

4.4.2 Sample Preparation for the X-Ray Diffractometer

The sample preparation technique used for the X-ray diffractometer depends on the amount of debris received. If the amount of particles is large, they can be mounted from the back of the Philips rectangular sample holder (3). The particles are first ground to less than 30 micrometers with an agate mortar. Before mounting, the powdered debris is then densely packed to reduce preferred orientation and increase the X-ray peak intensity.

If the amount of particles is small, the background sample holder provided by Philips is used. The background sample holder consists of a single crystal of alpha-quartz cut off 15 degrees to the C-axis. The X-ray beam impinged onto the prepared planes of the single alpha-quartz crystal produces no reflection or background. Figure 28 shows diffractographs of glass and background sample holders scanned from 6 degrees to 80 degrees, 20 minutes. The background sample holder shows a much lower background than the glass sample holder.

V-ANALYTICAL PROCEDURES AND RESULTS

5.1 Optical Microscopy:

Debris submitted to KSC's Microchemical Analysis Branch are first examined under stereo microscope to determine the general morphological features of the particles. Optical observation provides information on the size and texture of the debris. It also allows the investigator to estimate the amount of inorganic and metallic particles and organic particles and fibers. The metallic and inorganic particles are then classified into components on the basis of color and texture.

Sometimes it is necessary to crush debris to determine its optical properties, such as refractive indices and birefringence. Figure 29 illustrates crushing methods used for different sizes of samples. For small amounts of debris, the debris is placed on double-stick tape, covered with Scotch tape, and struck with a hammer. The tape minimizes the number of lost particles. Large amounts of debris can be crushed with an agate mortar or placed under a metal plunger which is struck with a hammer.

McCrone and Delly (4) include a comprehensive study of the use of OM and polarized light microscopy (PLM) for particle identification and describe the sophisticated identification techniques in detail. Debris characterization is based on certain properties that can be observed with PLM and that are typical for specific chemical compositions of debris. Important properties are transparency, opacity, color, refractive index, birefringence (Figure 30), size, shape and morphology of crystallites and crystalline and non-crystalline materials (Figure 31). These properties can be determined for individual phases of particles to a lower size limit of approximately 0.5 to 1 micrometer in diameter with PLM and OM techniques.

Transparency, opacity and the colors observed with transmission or reflectance illumination yield primary information about debris identity. Both the refractive index, which is related to the molecular weight of the compound, and the anisotropy of the refractive index, which appears in noncubic crystals, provide further information.

Undercrossed polars, the birefringence properties (Figure 30) of anisotropic crystals, will cause the crystals to display color. The color displayed depends on the thickness and difference in refractive indices for the two vector components of the polarized light in the crystals. With the aid of the Michel-Levy color chart (5), the birefringence of a crystal can be estimated.

Size, shape and morphology of the individual crystallites (Figure 31) are good features for identification, because certain species often appear in rather narrow size ranges, depending on the specific origin of a phase. Shape and morphology of particulates are closely related to the properties of materials such as fibrous materials, poorly crystallized cryptocrystalline and crystalline substances. The particular sizes of crystallites may also suggest a particular crystalline structure.

Particles larger than 30 micrometers are measured with an optical micrometer or scanning electron image. Linear extension values (Table 3) are then used to determine the average particle size. The linear extension values represent the actual diameter of regular particles and the projected or statistical diameter of irregular particles. The average values of several measurements from individual samples are taken to determine an overall average particle size.

The estimated volume percent of each phase or particle type is represented by the relative area occupied by that phase or particle type on a particular planar surface. The estimation is made by comparing the observed distribution of each phase or particle type with a standard visual estimation chart. The error associated with the visual estimation method has not been completely evaluated, but the work performed so far indicates that the error is about 5 volume percent for each phase or particle type.

Some types of debris can not be identified by optical microscopy. These include needle shaped crystals, which can not be isolated from surrounding debris for refractive index determination, fine grained materials, opaque inclusions and poorly crystallized materials. A combination of SEM with EDS (Section 5.2) and XRD (Section 5.4) provides a rapid means of identifying such debris.

5.2 Scanning Electron Microscopy with Energy Dispersive Spectrometry:

5.2.1 Scanning Electron Microscopy:

The scanning electron microscope (SEM) has unique capabilities for analyzing surfaces. It is analogous to the reflected light microscope. While the reflected light microscope forms an image from light reflected from the sample surface, the SEM uses electrons for image formation.

Electrons have much shorter wavelengths than light photons, and shorter wavelengths are capable of generating higher resolution information. Enhanced resolution permits higher magnification without loss of detail. The SEM produces images with a high depth of field, allowing SEM micrographs to maintain the three-dimensional appearance of textured surfaces. The maximum magnification of electron beam instruments is beyond 800,000X. Because of instrumental parameters, practical magnification and resolution are limited to about 75,000X (40 Angstroms) for a conventional SEM.

Debris mounted on carbon planchets or tape are usually coated with carbon in a vacuum evaporator or gold in a sputter coater to prevent charging. The coating material forms a nearly continuous film over the sample. A carbon or gold film of about 20 nanometers thickness is transparent to an electron beam. Figures 32 through 64 are selected SEM photomicrographs of debris collected from some of the areas listed in Table 1. The following table gives a figure number and description of each SEM photomicrograph and lists the figure number of its corresponding EDS pattern. This table may also be used as a reference guide for materials commonly found in investigations involving Space Shuttle systems.

Figure Number of SEM Photo	**Sample Description**	**Figure Number of EDS Pattern**
32	corundum particles	65
33	white aluminum oxides	66
34	silica coatings	67
35	black dense tile	68
36-39	fused tile	69
40	rocket fuel cell	
41	light grey primer	70
42	glass fiber	71, 72
43	glass fiber surface	73
44	metallics	74, 75
45	carbon steel	76
46-48	microballoons	
49-51	eccospheres	77
52, 53	MoS_2	78
54	muscovite	79
55	paint	80, 81
56	RTV	82
57, 58	rust	83
59, 60	salt	84
61	SiC	85
62	white materials	86, 87
63, 64	zinc primer	88

5.2.2 Energy Dispersive Spectrometry:

For analysis by energy dispersive spectrometry (EDS), the debris samples are usually not coated with carbon or gold to avoid overlapping of X-ray peaks. The characteristics of X-rays generated by a sample are used to determine its elemental composition. The EDS analysis results are displayed as a spectrum of counts versus energy.

As each X-ray enters the silicon crystal detector, it produces a shower of photoelectrons that strikes the silicon atoms and excites bound electrons to the conduction band. The total number of freed electrons is linearly proportional to the energy of the entering X-ray. The charge is collected by an applied bias voltage before the next X-ray enters the detector. The charge is amplified by a field-effect transistor (FET) preamplifier with a stairstep of voltage output. The height of each step is proportional to the energy of the incident X-ray.

Carbon (C) and Oxygen (O) can be detected by EDS. The elemental composition of most debris is listed in order of decreasing abundance (Table 3). The order is based on peak intensities recorded during analysis where the peak intensity is roughly proportional to the element concentration. Figures 65 through 88 are the EDS patterns of the particulates from the locations listed in Table 1. The elemental data from EDS is used as an aid for phase identification by XRD.

For accurate elemental identification and concentration determination, it is imperative that the system energy calibration be set correctly. The calibration for KSC's Microchemical Analysis Laboratory is performed after installation or during any maintenance of the X-ray detector, preamplifier or amplifiers.

5.3 Analytical Electron Microscopy with Wavelength Dispersive Spectrometry:

5.3.1 Analytical Electron Microscopy:

The analytical electron microscope (AEM) is a microanalytical tool that combines light microscopy (reflected light) with SEM and WDS. Samples as small as 1 cubic micrometer can be analyzed, and detection limits are as low as 0.001 weight percent for nearly all elements. The main differences between AEM and SEM are that AEM requires a higher beam intensity and a higher voltage, and for AEM the specimen stage is fixed with a fixed take-off angle. Beyond these small differences, the principles and techniques of AEM are so similar to those of SEM that it would be redundant to describe them further.

5.3.2 Wavelength Dispersive Spectrometry:

The wavelength dispersive spectrometer (WDS) can detect elements with atomic numbers in the range of 4 (beryllium) to 94 (Plutonium). This report documents WDS analysis of samples having flat surfaces and sizes greater than 30 micrometers. Analysis techniques for rough surfaces, particles and biological specimens are less advanced and are still under development.

For analysis by WDS, debris are usually polished and coated with carbon. When a beam of high energy electrons strikes the sample, X-ray spectra characteristic of the elements present are generated. An X-ray line is the result of an electron shift from an outer shell into inner K, L or M shells.

5.3.2.1 Qualitative Analysis:

Qualitative analysis by WDS is based on the ability of a spectrometer system to measure characteristic line energies and relate them to the presence of specific elements. The characteristic X-ray spectra constitute "fingerprints" for identification of the elements.

The resolution of the wavelength dispersive spectrometer, being typically less than 10 electron volts (ev), is much better than the resolution of the energy dispersive spectrometer, at 150 ev. The better resolution of WDS leads to a peak-to-background ratio at least 10 times higher than that of EDS. As a result, more members of the family of X-ray lines can be detected, making WDS useful for positive detection and identification of elements at low concentrations. Comparing the EDS spectrum (Figure 89) with the WDS spectrum (Figure 90) for the L-alpha lines of molybdenum and the K-alpha lines of sulfur shows that many of the peaks resolved in the WDS spectrum are not detected in the EDS spectrum. The higher resolution of WDS allows the separation of almost all peak overlaps that occur in EDS.

5.3.2.2 Quantitative Analysis:

Quantitative analysis by WDS involves accurate measurement of the intensity of the spectral lines corresponding to preselected elements for both samples and standards under identical operating conditions. From the spectral line intensity measurements, intensity ratios are calculated. The ratios are then converted into chemical concentrations using the ZAF (Z: atomic number, A: absorption, F: fluorescence) correction method to further improve the accuracy of the analysis. It should be pointed out that the ZAF technique is still being improved, especially for low electron energies and light element analysis.

The analytical results of three replicate analyses of NBS standards K-412 and K-497 are listed in Tables 5 and 6, respectively. The analytical results for the observed mean and standard deviation of the observed mean are in agreement with the actual weight percent and standard deviation of the actual weight percent.

Note that the standard deviations of the observed mean for magnesium (Table 5), oxygen (Tables 5 and 6) and phosphorous (Table 6) are large compared to those of the other listed elements. The higher standard deviations are probably due to the rougher surfaces and the higher absorptions of these three elements. It may be possible to improve precision by improving the control of factors such as sample preparation, instrumental processes and probe current levels.

Table 5. Electron Microprobe Analysis of NBS STD K-412 with Observed Mean and Standard Deviation

Element	Actual Weight %	1st Run Wt. %	2nd Run Wt. %	3rd Run Wt. %	Observed Mean	Std Dev Actual Weight %	Std Dev Obs. Mean
Si	21.06	20.90	21.46	21.20	21.19	0.16	0.28
Fe	7.74	7.49	8.20	7.32	7.67	0.27	0.47
Mg	11.66	10.56	12.40	13.59	12.18	1.63	1.53
Ca	10.88	10.03	10.59	9.89	10.17	0.94	0.37
Al	4.91	4.48	4.86	4.36	4.57	0.49	0.26
O	43.75	45.95	43.53	40.83	43.44	2.59	2.56
Total	100.00	99.41	101.09	97.25	99.25	Avg 2.10	1.92

Table 6. Electron Microprobe Analysis of NBS STD K-497 with Observed Mean and Standard Deviation

Element	Actual Weight %	1st Run Wt. %	2nd Run Wt. %	3rd Run Wt. %	Observed Mean	Std Dev Actual Weight %	Std Dev Obs. Mean
Si	0.13	0.09	0.18	0.10	0.12	0.07	0.049
Pb	0.92	0.08	0.89	0.85	0.61	0.60	0.460
P	33.18	34.32	30.93	30.73	31.99	2.49	2.070
Mg	5.21	5.30	6.93	5.60	5.94	1.24	0.870
Al	5.78	5.37	6.00	5.19	5.52	0.34	0.420
B	0.04	-	0.28	-	0.09	-	-
Zr	0.40	0.30	0.56	0.51	0.46	0.16	0.140
Ti	0.21	0.13	0.18	0.23	0.18	0.07	0.049
Ce	0.62	0.50	0.59	0.40	0.50	0.19	0.090
Ta	0.80	0.90	1.51	0.80	1.07	0.50	0.380
Fe	0.24	0.20	0.28	0.31	0.26	0.07	0.056
Li	0.001	-	-	-	-	-	-
O	52.47	52.10	51.63	54.40	52.71	1.51	1.480
Total	100.00	99.29	99.99	99.63	99.64	Avg 0.56	0.350

To demonstrate the magnitude of the discrepancies between measured values and true values, the standard deviation of each element from the actual percent was calculated. If it is assumed that the samples are well prepared and represent true values, then the standard deviations from the actual percent will represent the degree of accuracy. As noted in the case of the observed mean, the standard deviations from the actual mean for magnesium, oxygen and phosphorus are higher than those of the other elements. Again, this is probably caused by their higher absorption of X-rays.

Since quantitative analysis can now be performed with relatively high accuracy, great care must be taken to ensure that the measured response of the X-ray detector system is linear over a wide range of counting rates, and that the useful signal can be easily extracted from the background. The accuracy of the analysis is dependent on standards and operating conditions such as current, operating voltage and X-ray lines.

5.4 X-Ray Diffraction:

The analytical techniques of X-ray diffraction (XRD) can only be applied to crystalline materials. The principle is based on the fact that crystalline materials are made up of a regular array of atoms. The atoms form planes which have various orientations to specific crystal axes. These planes form a regular pattern of orientations and the distances between them, called interplanar spacing. The wavelengths of X-rays have the same order of magnitude as the distances between the planes and can be constructively reflected from them, forming a unique X-ray pattern or signature for each crystalline material.

With XRD, only the phases present can be identified, and nothing can be said about their textural relationships. Therefore, identification of the source of the debris is not automatic, and a preliminary study by OM is often necessary. A combination of OM and XRD are used for otherwise intractable debris problems.

The two types of instrumentation used for X-Ray diffraction are the X-ray powder film camera and the X-ray diffractometer. The objective of both instruments is to measure the intensity of diffracted X-rays with respect to some reference angle (theta) of a particular atomic plane. Qualitative phase identification is achieved using the position of two theta and the intensity of the X-rays.

5.4.1 X-Ray Powder Film Camera:

In a powder film camera, the film is exposed in a light-tight enclosure where the X-rays reflected from the planes cause darkening in the form of lines on the film surrounding the sample. The sample can be as small as a few milligrams. The intensity of each line is the estimated value of its darkness. Errors in estimation of intensity do not significantly affect the qualitative search and match for phase identification.

There are various sources of error for the powder film camera. Radius error is the error produced by the difference between apparent and true values of the theta angle.

Specimen eccentricity is caused by faulty assembly of the camera, causing the rotation axis of the camera to be displaced from the rotation axis of the film-supporting structure. Horizontal and vertical divergence of the beam is related to the position of the slit, and its contribution to the error is negligible. Other sources of error are film shrinkage, absorption of the X-ray beam by the specimen and mistakes made while reading the scale.

The three principle approaches for correcting errors are the use of calibration standards, refinements in experimental techniques and mathematical procedures. In this study, calibration standards were used. A substance possessing a known lattice constant was used to calibrate the film. With this technique, the accuracy of one part per thousand in the position of two theta was maintained.

5.4.2 X-Ray Diffractometer:

The X-ray diffractometer is a mechanical goniometer where the sample turns at some angle theta, and the detector moves at twice theta to intercept the diffracted X-ray beam. The accurate position of two theta (d-spacing) is affected by the alignment of the goniometer. The Microchemical Analysis Branch tries to maintain an accuracy of one part per thousand in the position of two theta by aligning the goniometer at least every six months. With this level of alignment accuracy, most crystalline compounds, including solid solution series, can be identified.

The intensity of the diffracted lines can be affected by grinding, particle size, crystallinity, packing density, microabsorption and preferred orientation (3). Of these, the single most important factor limiting the accuracy of the intensity appears to be preferred orientation. The samples in this study were packed from the back of the sample holder against a flat, rough surface, which was later removed. This packing technique minimizes the reduction of the intensity.

VI-CONCLUSIONS AND RECOMMENDATIONS

The combination of optical microscopy (OM), scanning electron microscopy (SEM) with energy dispersive spectrometry (EDS), analytical electron microscope (AEM) with wavelength dispersive spectrometry (WDS) and X-ray diffraction (XRD) has proven to be the most effective approach to debris characterization.

OM techniques were successfully used to determine particle sizes, glass phases and amorphous phases, to characterize the morphology and size of crystallites and poorly crystallized materials and to estimate the volume percent of each component. The identification of very small anisotropic compounds was also successfully accomplished using OM.

SEM with EDS provided information on the surface morphology and qualitative elemental content of debris and was used to aid in XRD phase analysis. AEM with WDS was used to determine the detailed surface morphology and quantitative elemental content of debris. XRD was used to identify the phases of materials.

A follow-up study should be performed to determine the precision and accuracy of the estimations of volume percent and particle size of each component by OM. Criteria for the linear extension values for particle sizes should be established. The accuracy and precision of the estimated volume percent of each component can be improved by comparing the observed distribution of each phase with the standard visual estimation chart.

The success and relative accuracy of quantitative analysis by EDS and WDS depends largely on specimen preparation and obtaining good peak-to-background ratios for the characteristic X-ray lines. Specimen preparation is the direct responsibility of the experimenter, while good peak-to-background ratios result from good operation of the analytical instruments. Statistical analysis of X-ray counts for the various sample preparation techniques should be performed. Optimization of peak-to-background ratio by counting X-ray spectral lines could be developed laboratory for better counting statistics.

Optimum quantitative phase analysis techniques for XRD should be developed. Further studies should be performed on the effects of microstrain due to particle size reduction, line broadening due to cryptocrystallinity, preferred orientation of the platy minerals and microabsorption effects. The major contributor to high standard deviation appears to be the preferred orientation of the platy minerals. Techniques developed to limit preferred orientation, such as the spray-dry technique and the dilution method, should be considered.

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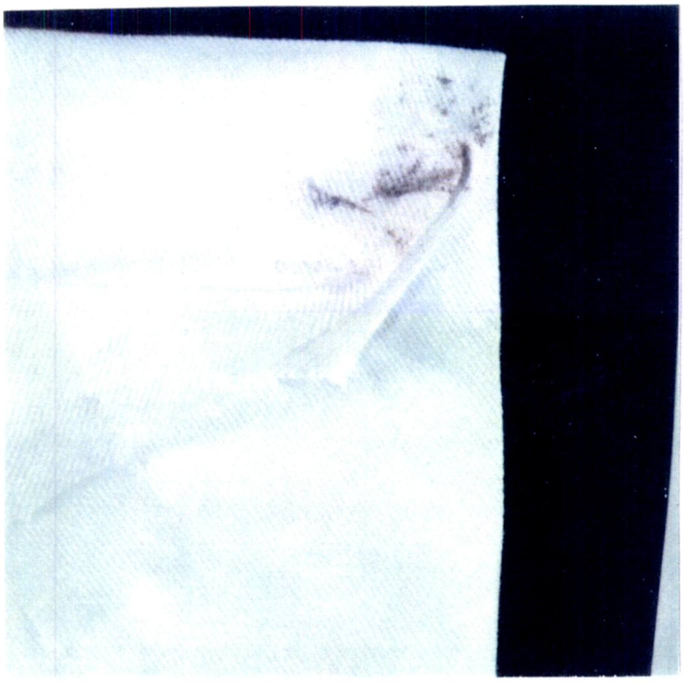


Fig. 1
Particles on lint-free cloths 1.4X

Fig. 2
Debris on Texwipe 1.1X

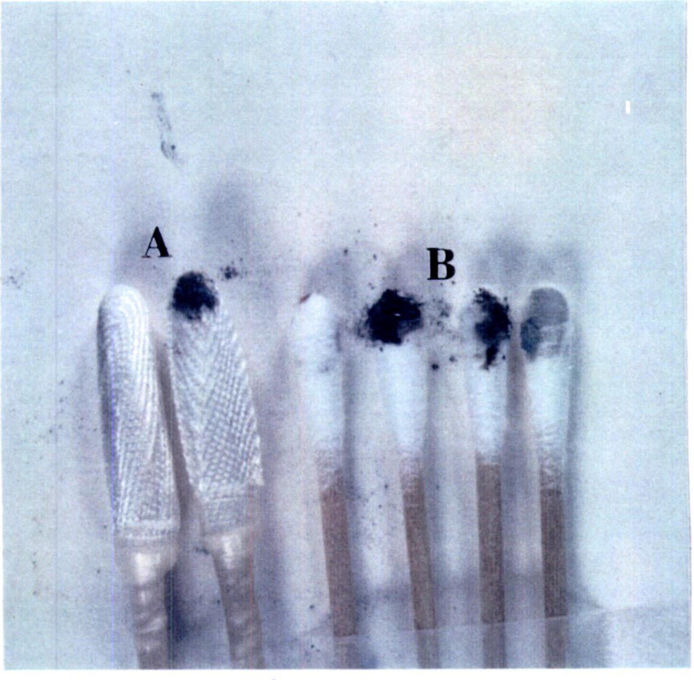


Fig. 3
Particles on organic (A) and cotton (B) swabs 1.4X

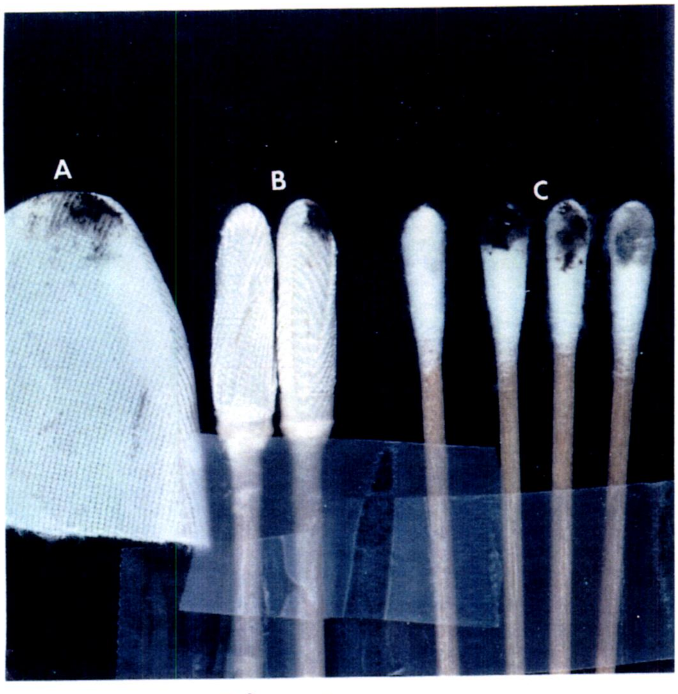


Fig. 4
Particles on organic glove finger (A), organic swab (B) and cotton swab (C) 1.4X

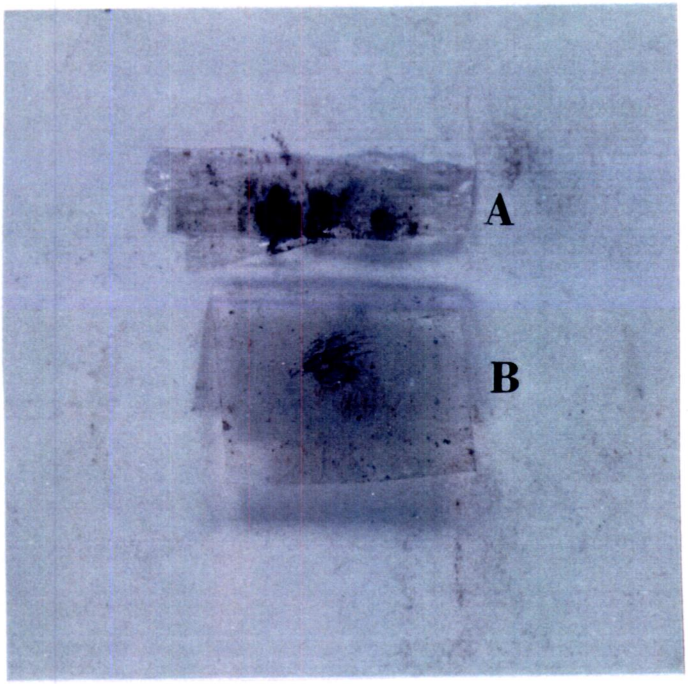


Fig. 5
Particles on double (A) and single (B) stick tape 1.7X

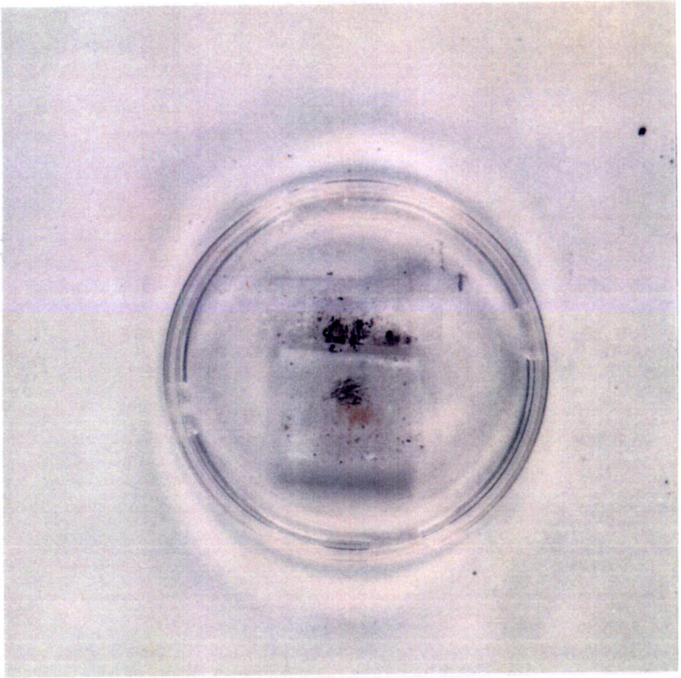


Fig. 6
Particles on Scotch tape in petri dish 1X

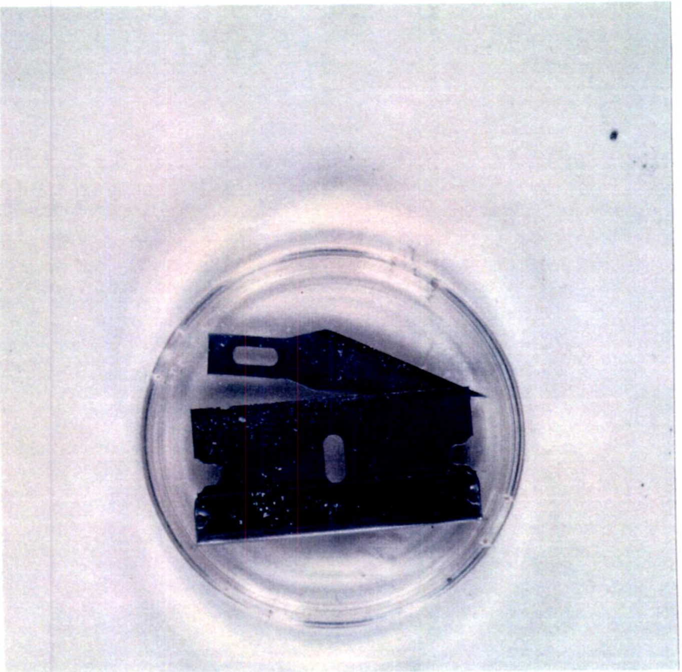


Fig. 7
Particles collected on razor blade edge 1X

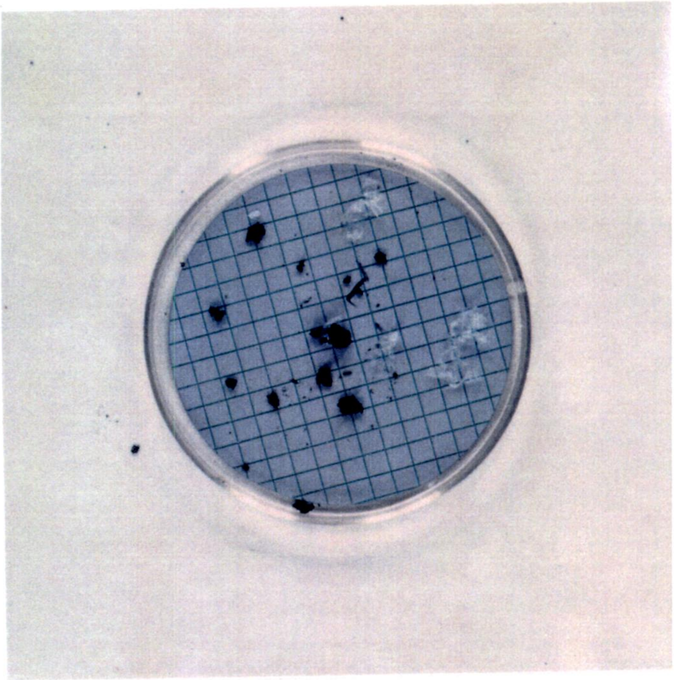


Fig. 8
Particles collected on Millipore filter paper 1X

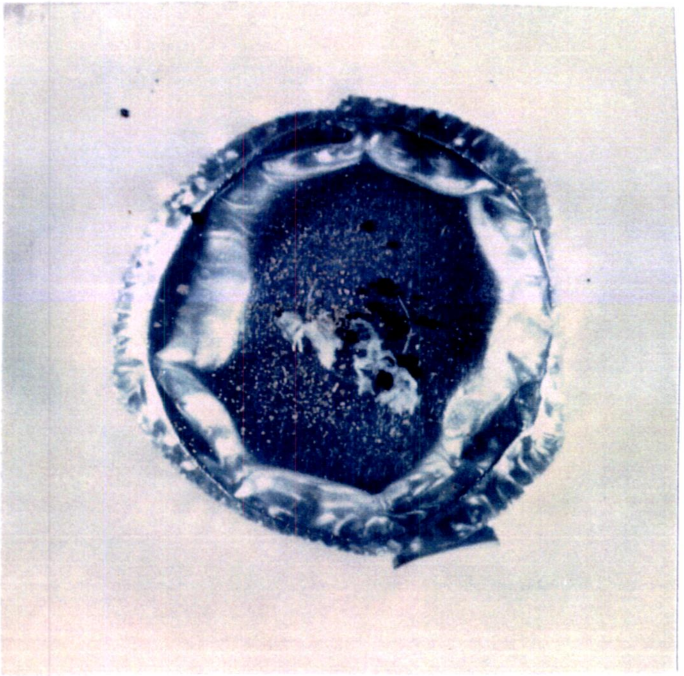


Fig. 9
Non-volatile residue (NVR) on Al dish 1X

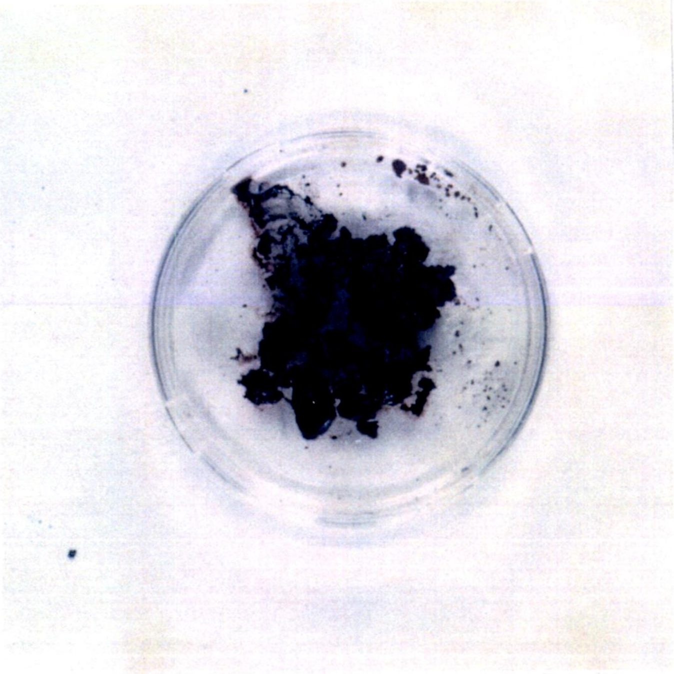


Fig. 10
Sediment collected on clear petri dish 1X

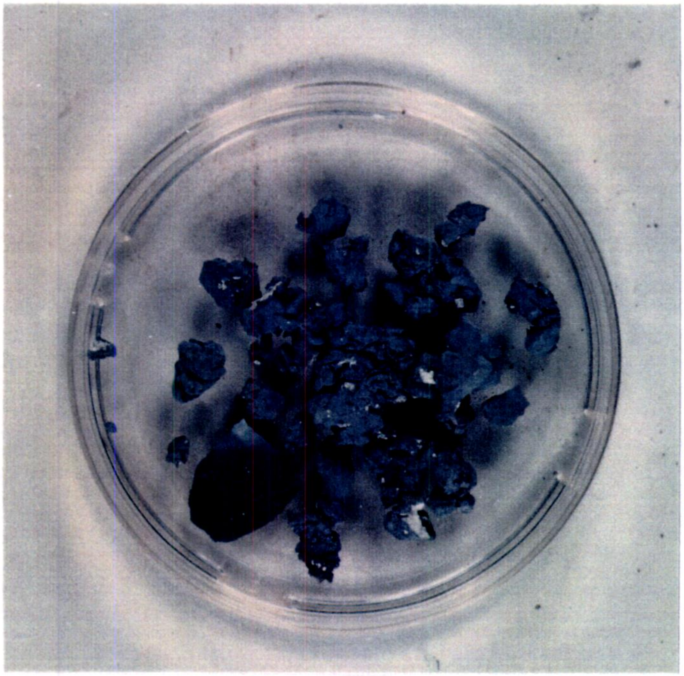


Fig. 11
Zn primer in petri dish 1.4X

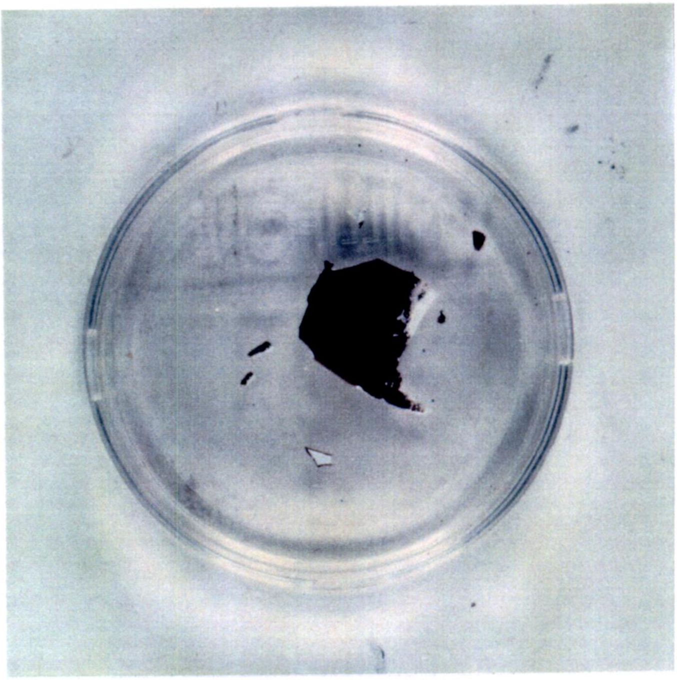


Fig. 12
Room Temperature Vulcanizing materials (RTV) in petri dish 1.2X

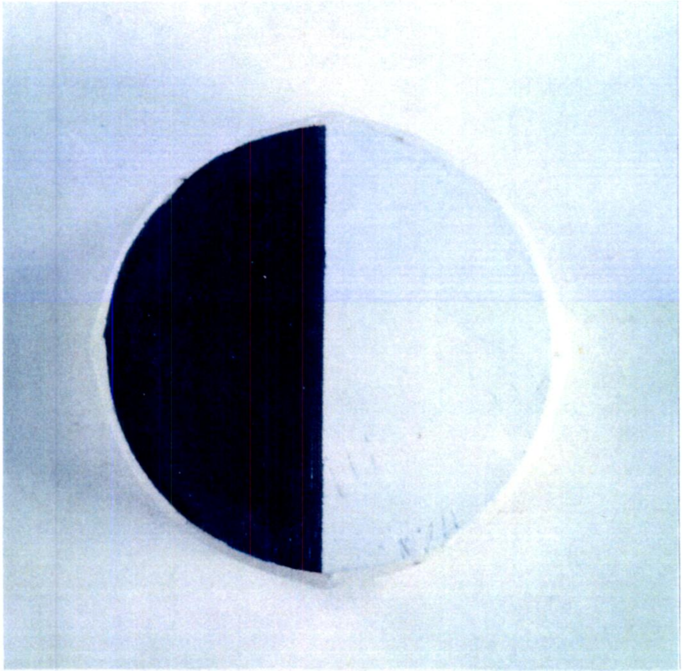


Fig. 13
Special sample preparation base with black and white background 1.2X

Fig. 14
Special sample preparation base with samples 1.2X

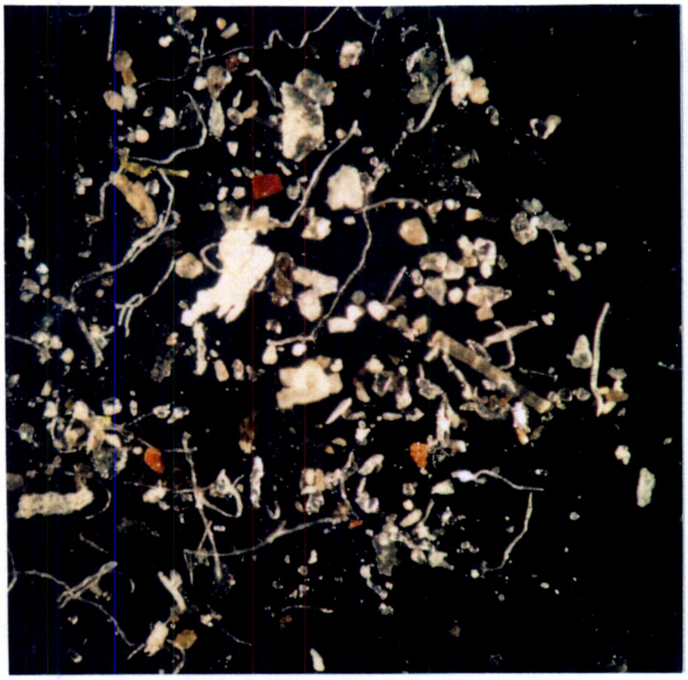


Fig. 15
Particulates on black background 22X

Fig. 16
Particulates on white background 22X

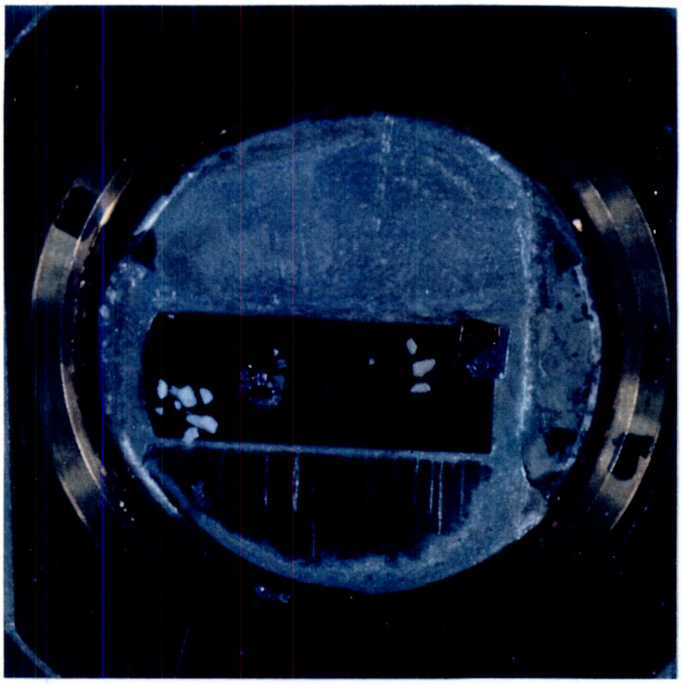


Fig. 17
Particulates mounted on
Be block, SEM specimen
holder 2.6X

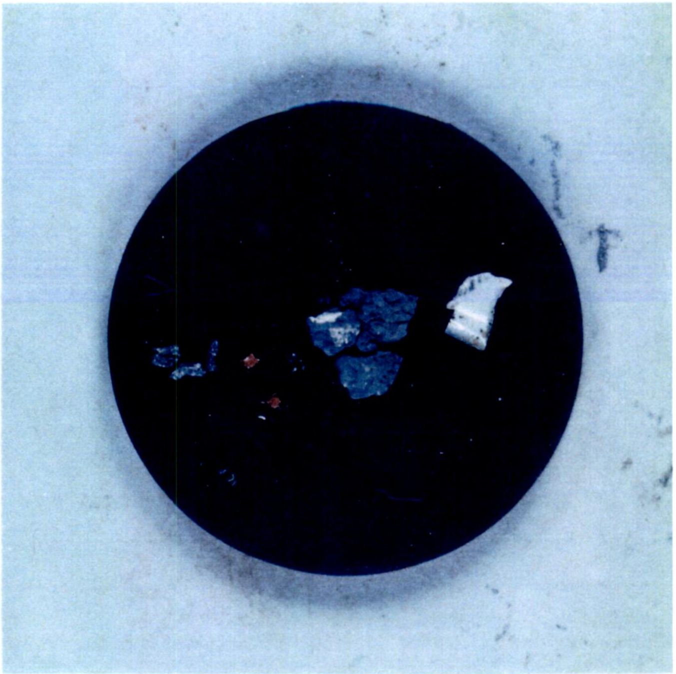


Fig. 18
Particulates mounted
on carbon planchet
2.5X

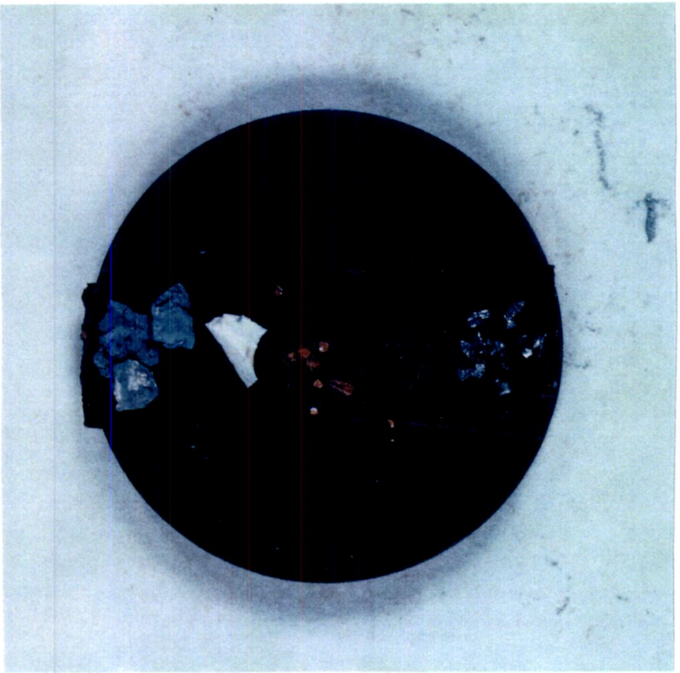


Fig. 19
Particulates mounted by
double stick carbon tape
to carbon planchet 2.5X

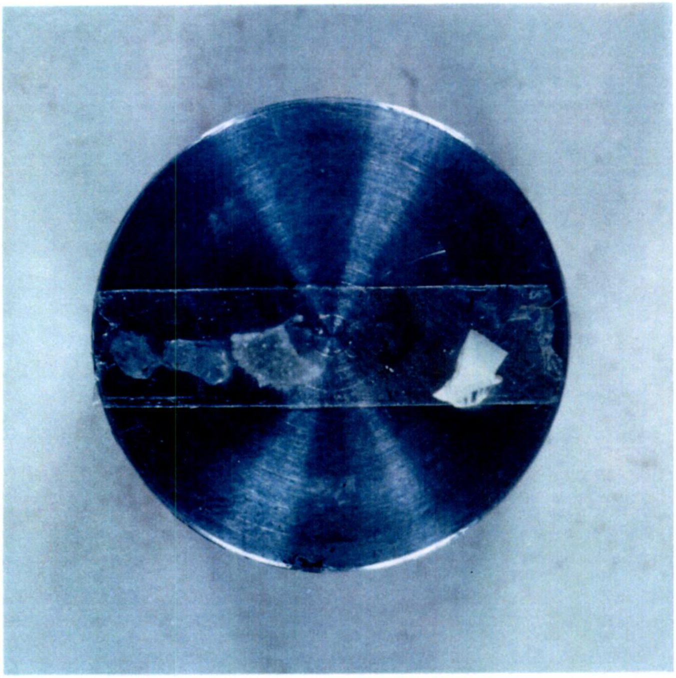


Fig. 20
Particulates mounted
by double stick clear
tape to Al block 2.6X

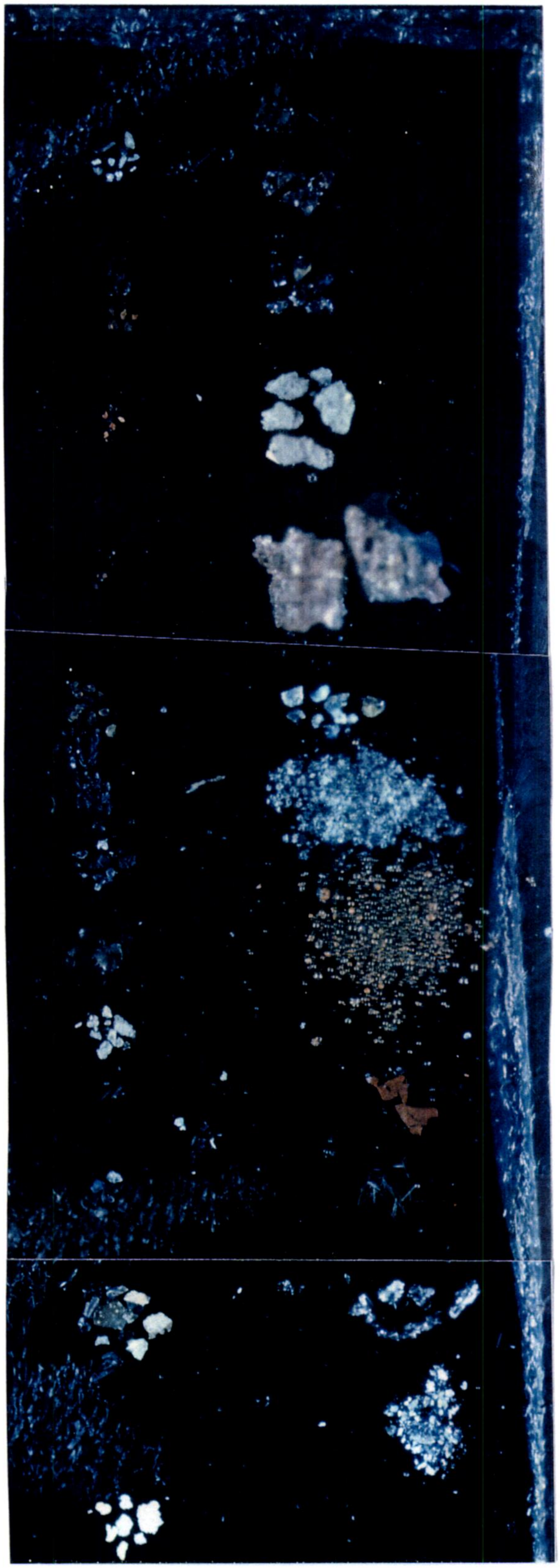


Fig. 21 Particles mounted on double stick carbon tape 10X

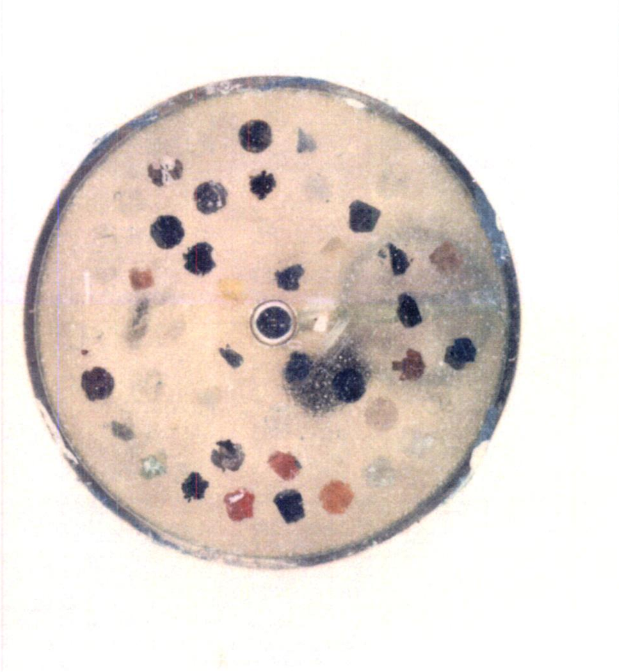


Fig. 22
Large debris standard samples mounted with resin and polished 5X

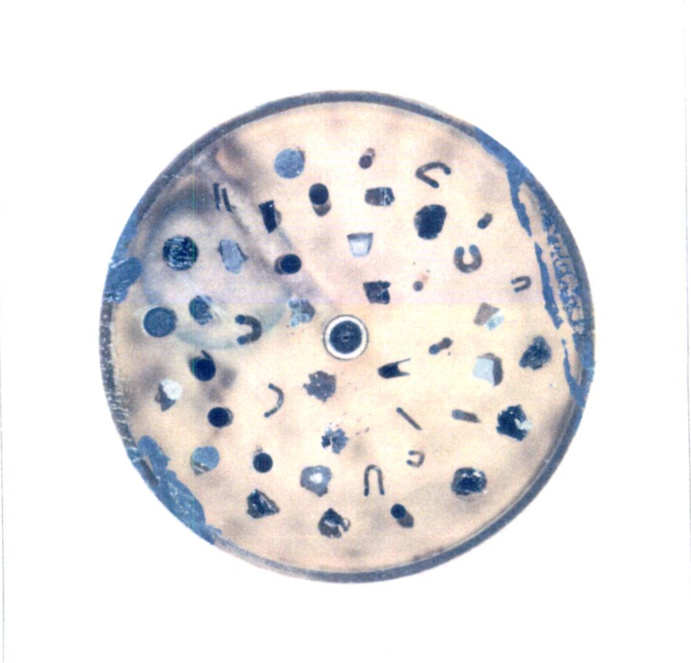


Fig. 23
Large metal particles mounted with resin and polished 5X

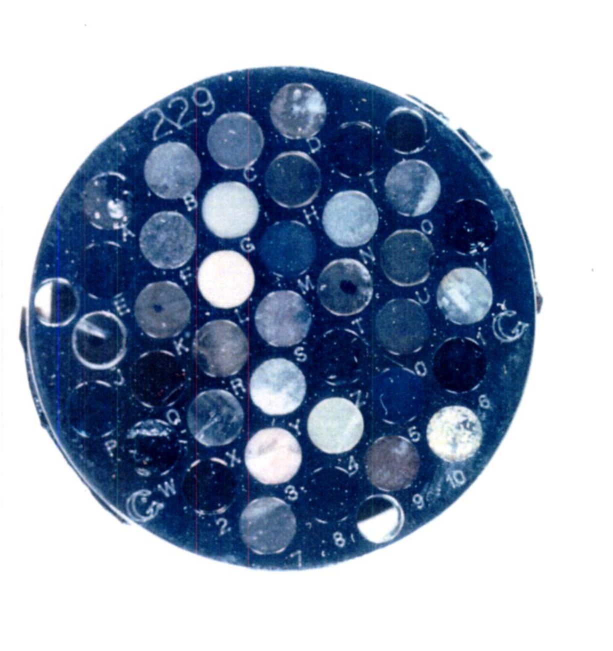


Fig. 24
Individual particles polished and mounted mechanically 5X

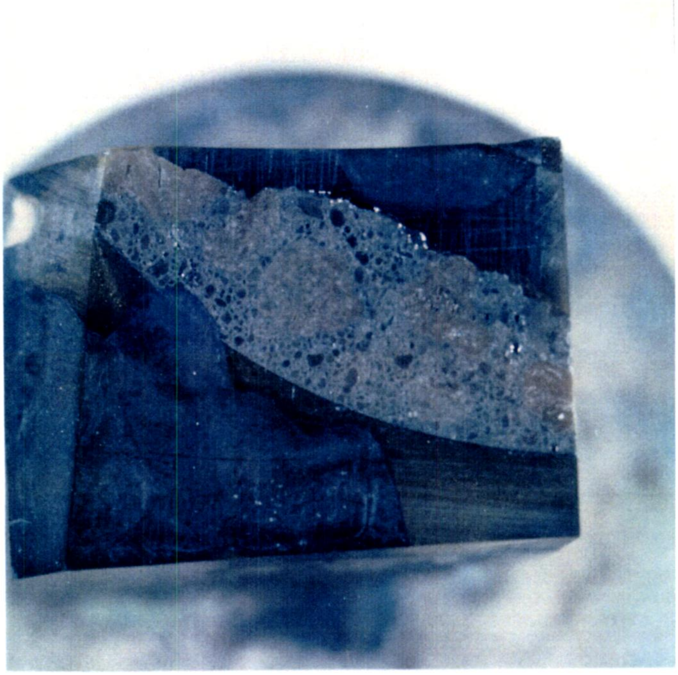


Fig. 25
Resin impregnated concrete polished for microprobe analysis 5X

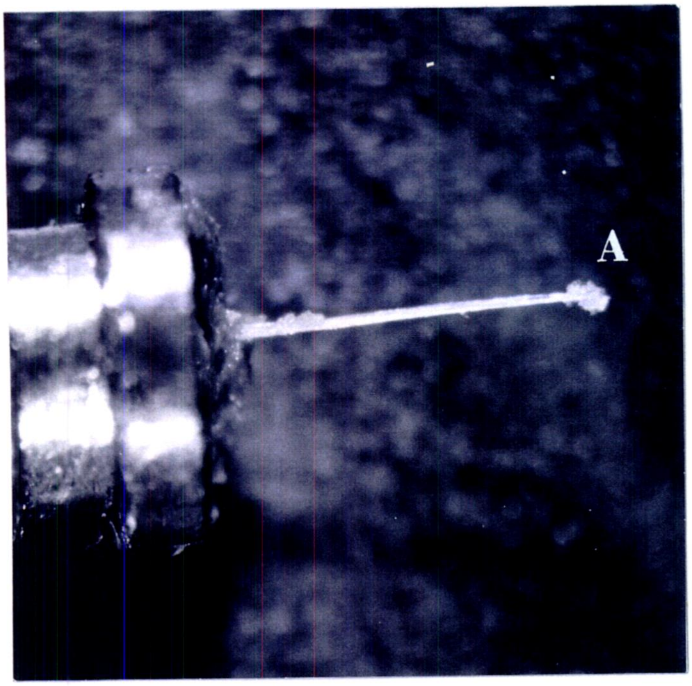


Fig. 26
Glass fiber with brass supporter and particles at glass fiber end (A) 17X

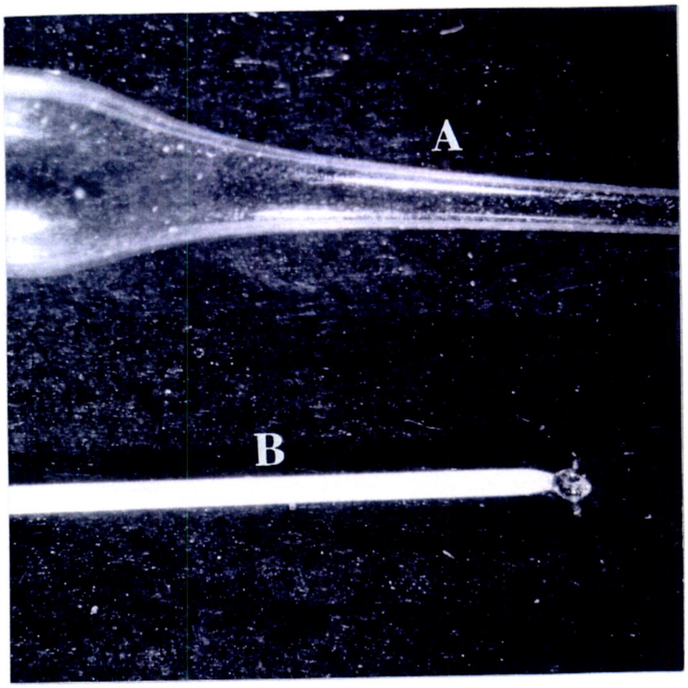
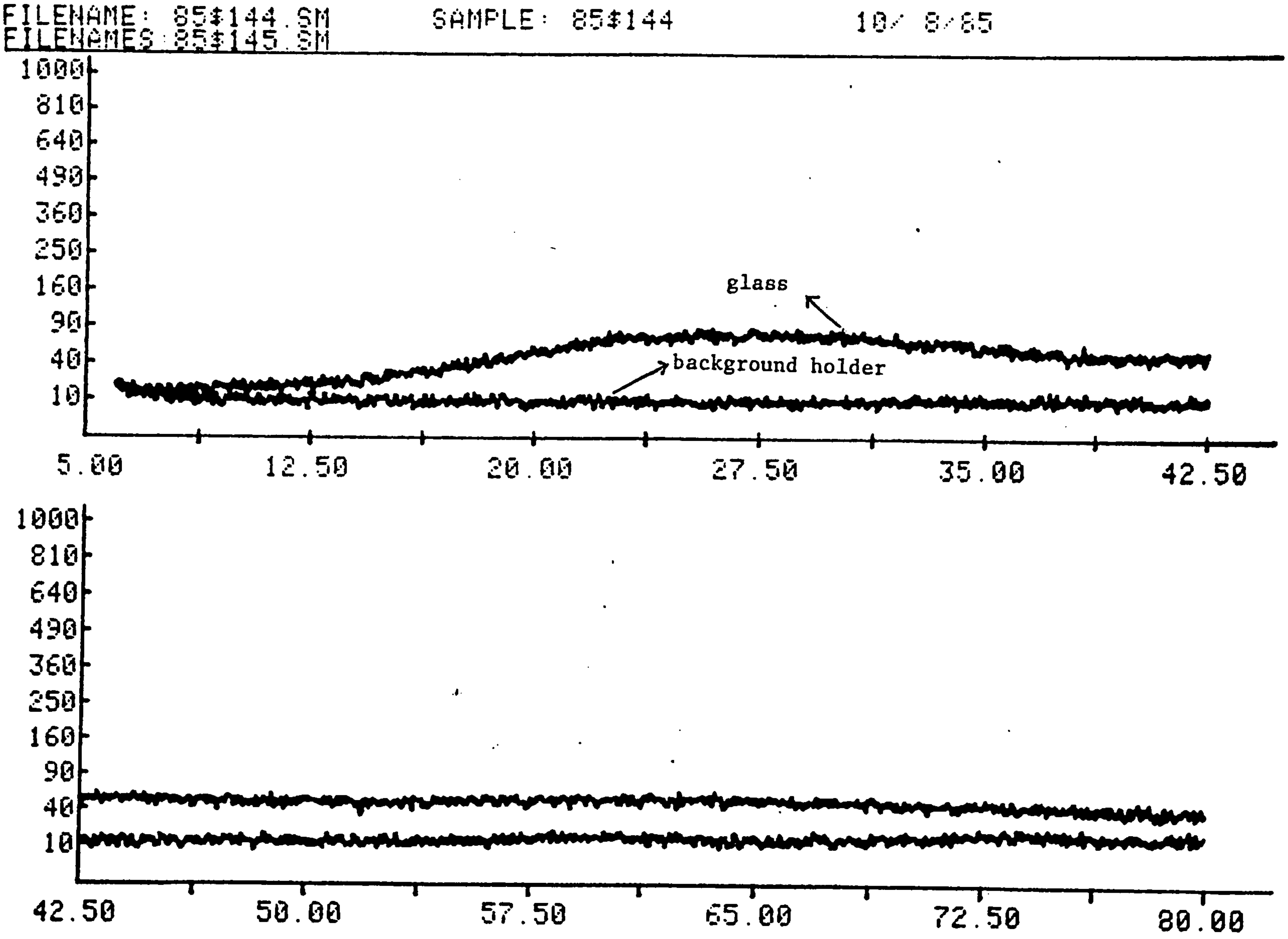
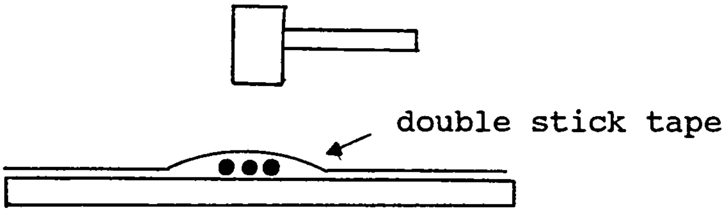


Fig. 27
Glass capillary tube for X-ray film camera, blank (A) and packed with sample (B) 17X

Fig. 28 Diffractograph of glass and background sample holders

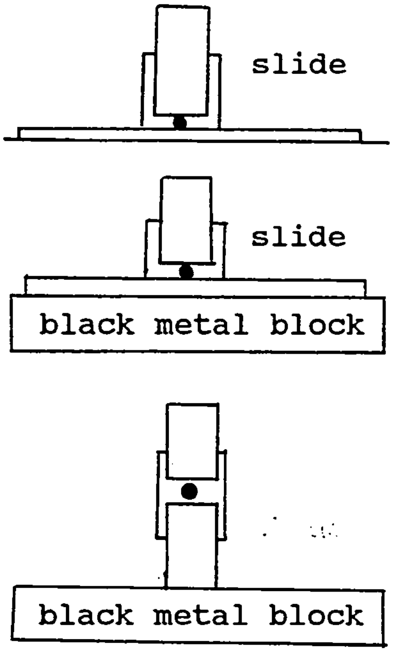


(1) Crushing a small amount of debris



Cover the debris with double stick tape, and break it by striking it with a hammer.

(2) Crushing a small amount of debris



A. Easy to crush: hand pressure

B. Hard to crush: gentle strike with a hammer

C. Very difficult to crush: strike with a hammer

Fig. 29 Particle crushing methods

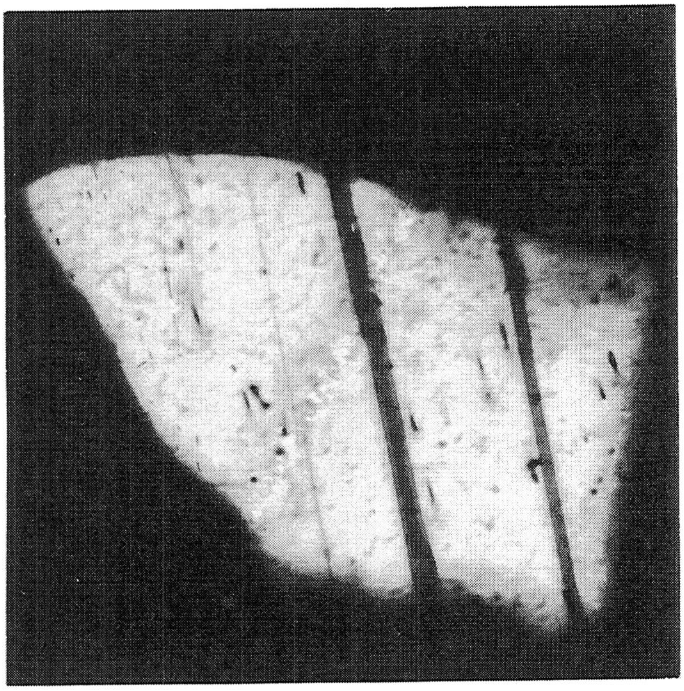


Fig. 30
Albite crystals ($NaAlSi_3O_8$)
polarized light 530X

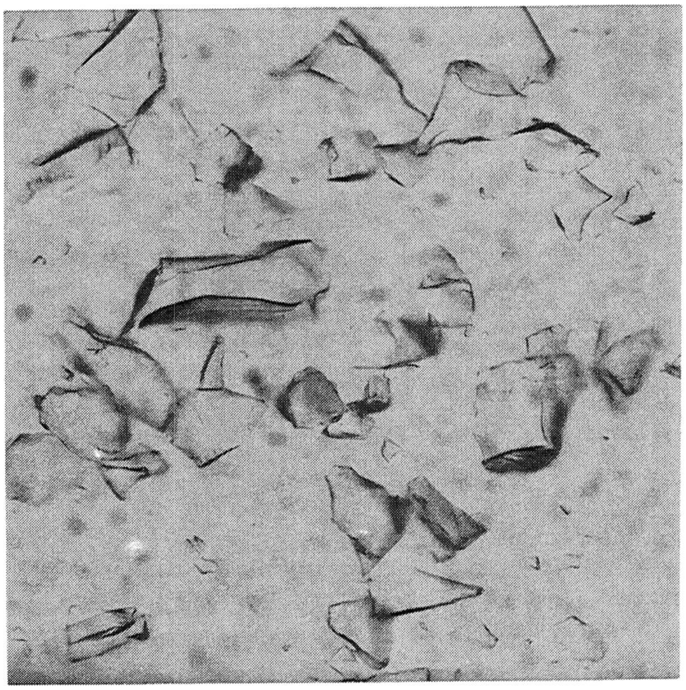


Fig. 31
Amorphous glass particles, polarized light
137X

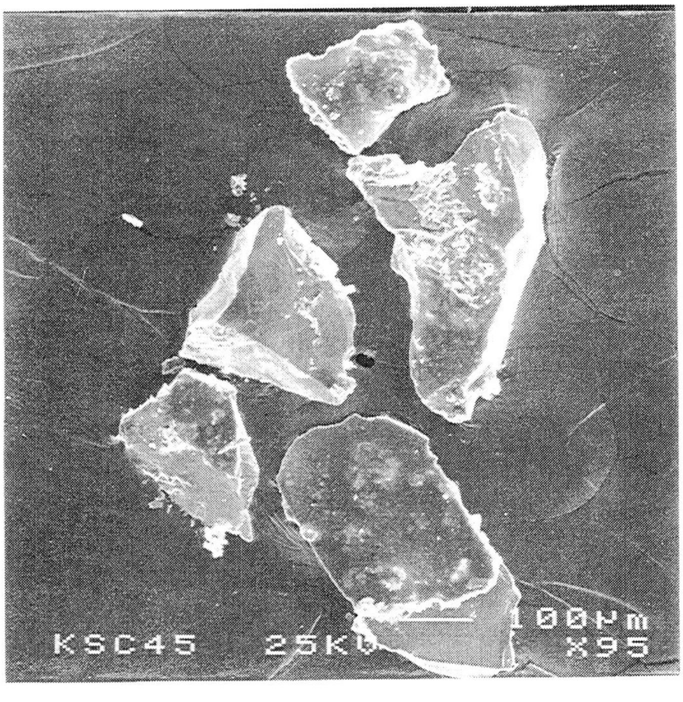


Fig. 32
Corundum particles
95X

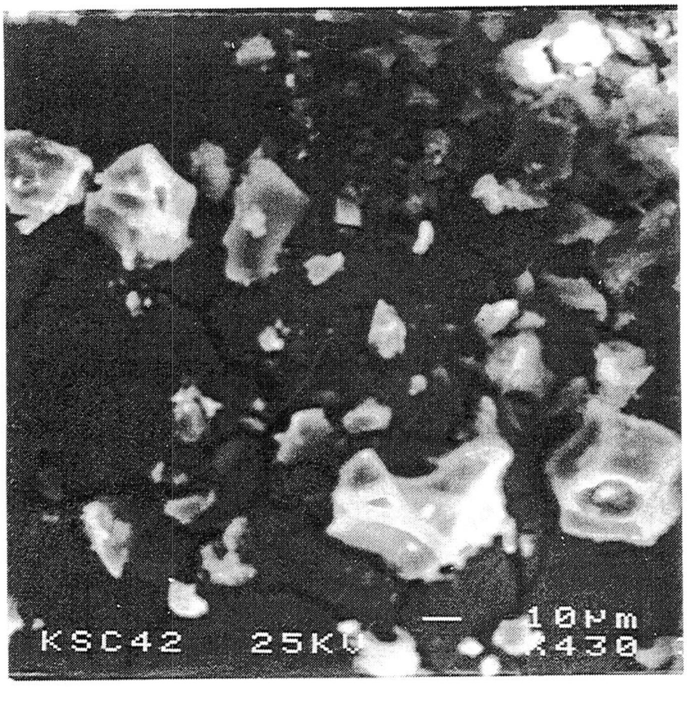


Fig. 33
White aluminum oxides
430X

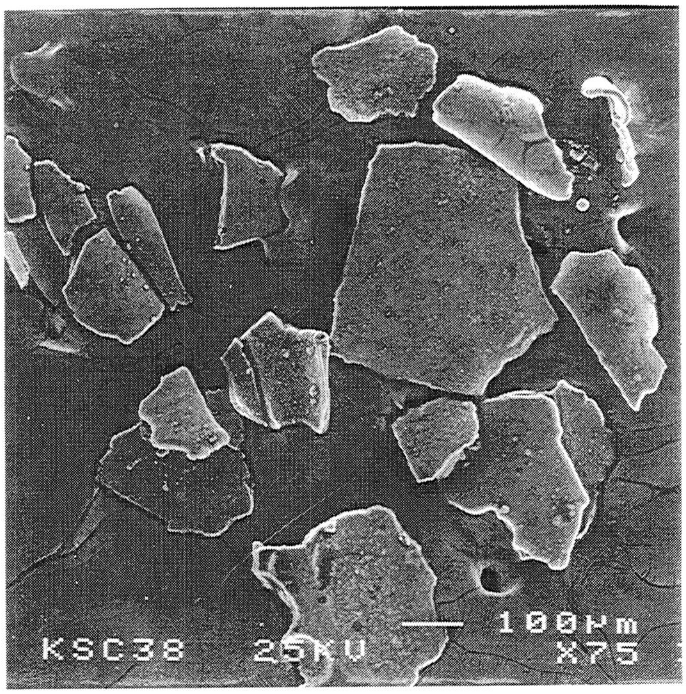


Fig. 34
Silica coatings 75X

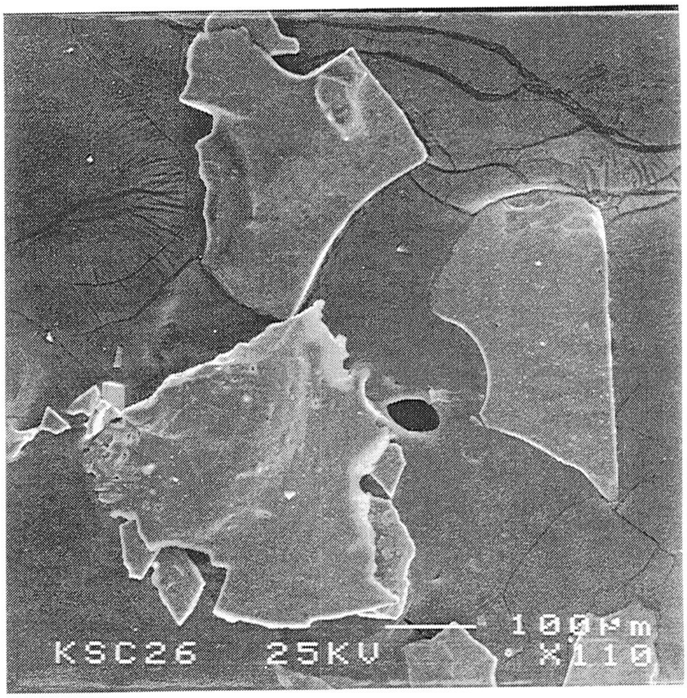


Fig. 35
Dense black tile 110X

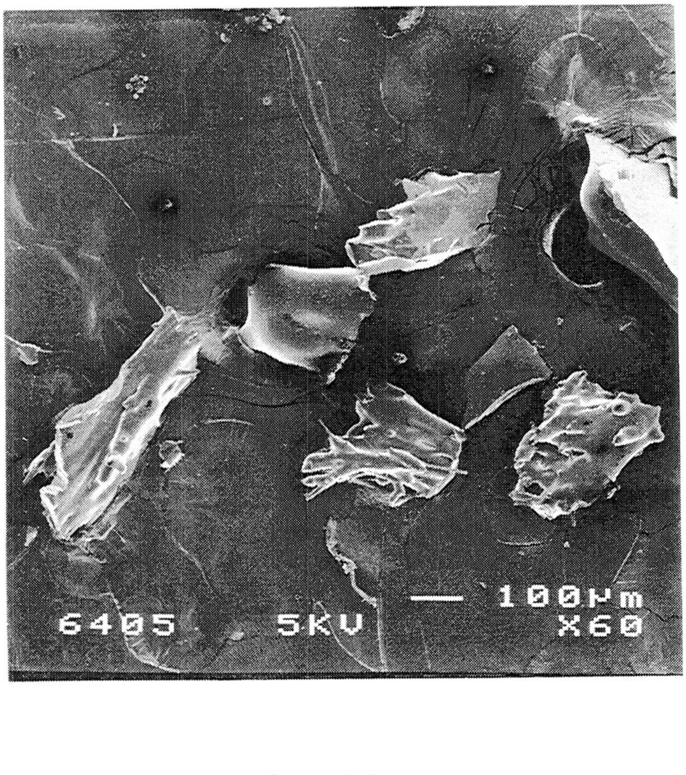


Fig. 36
Fused tile 60X

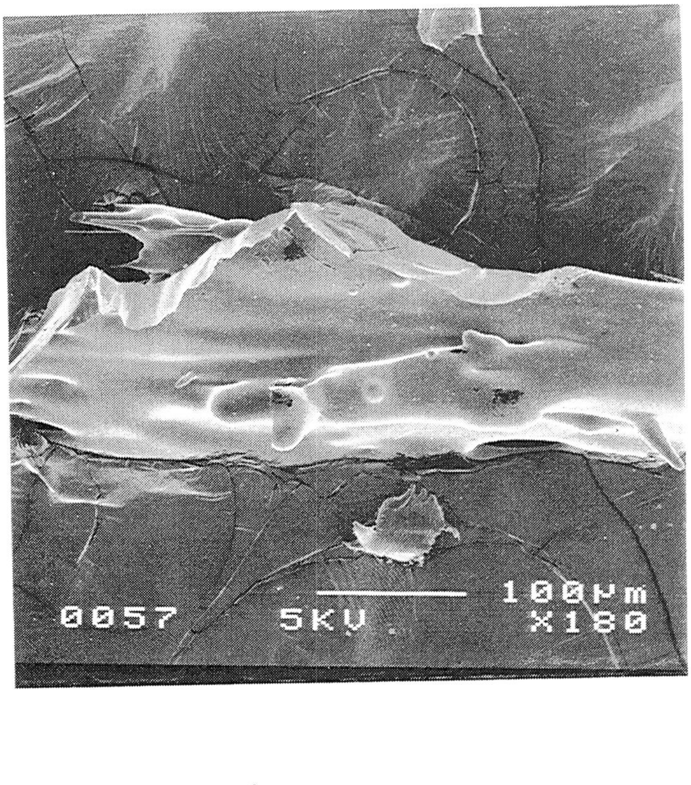


Fig. 37
Fused tile 180X

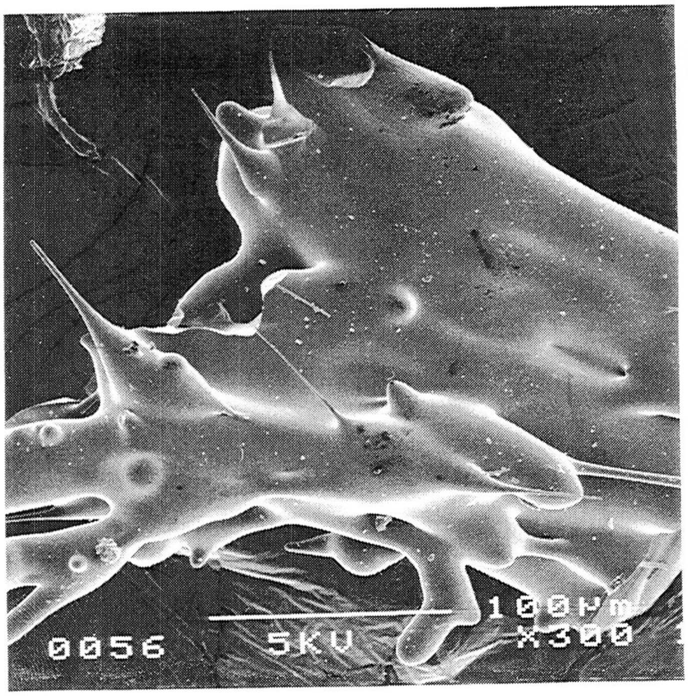


Fig. 38
Fused tile 300X

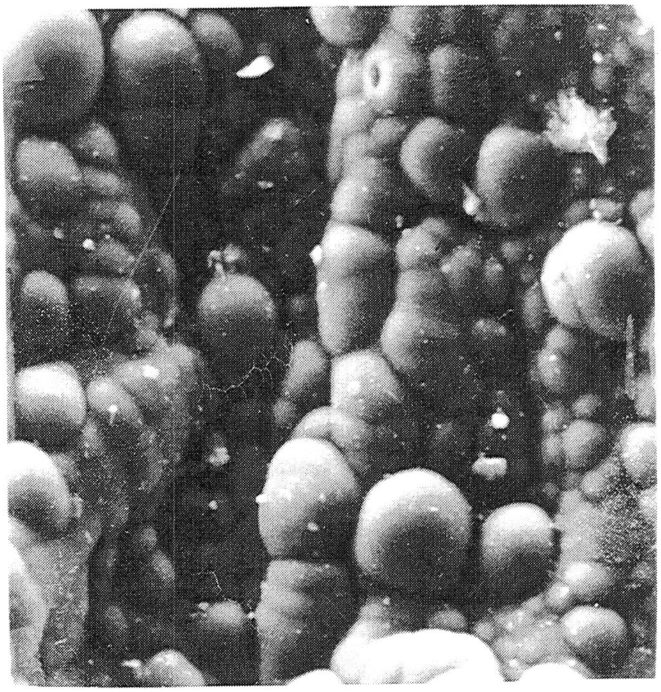


Fig. 39
Fused tile surface
540X

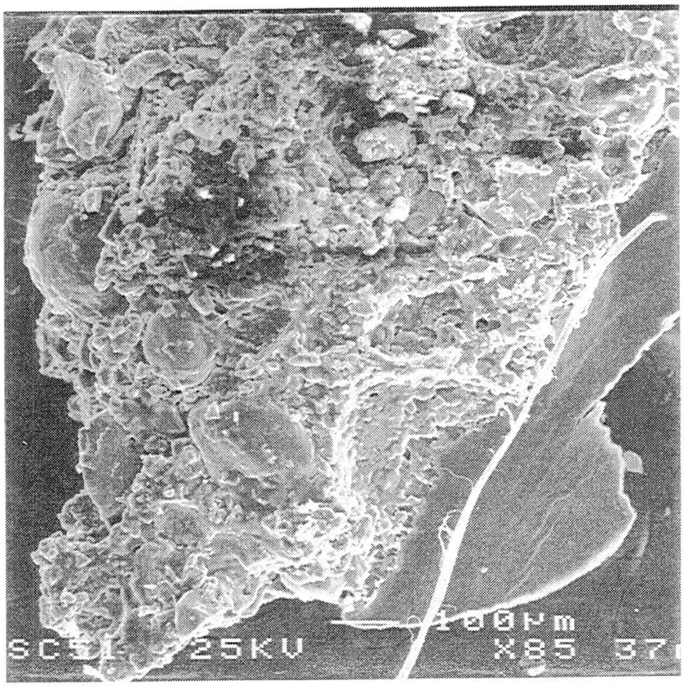


Fig. 40
Rocket fuel cell
(Al + NH_4Cl) 85X

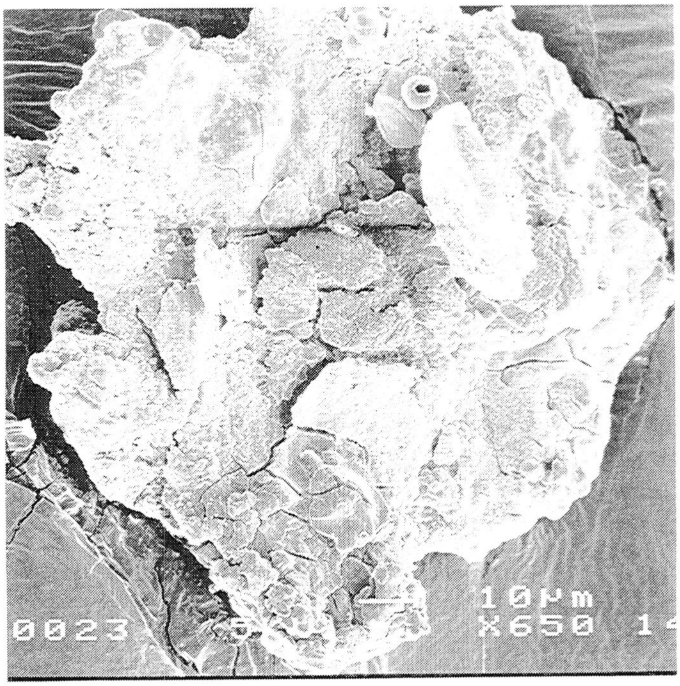


Fig. 41
Light grey primer
650X

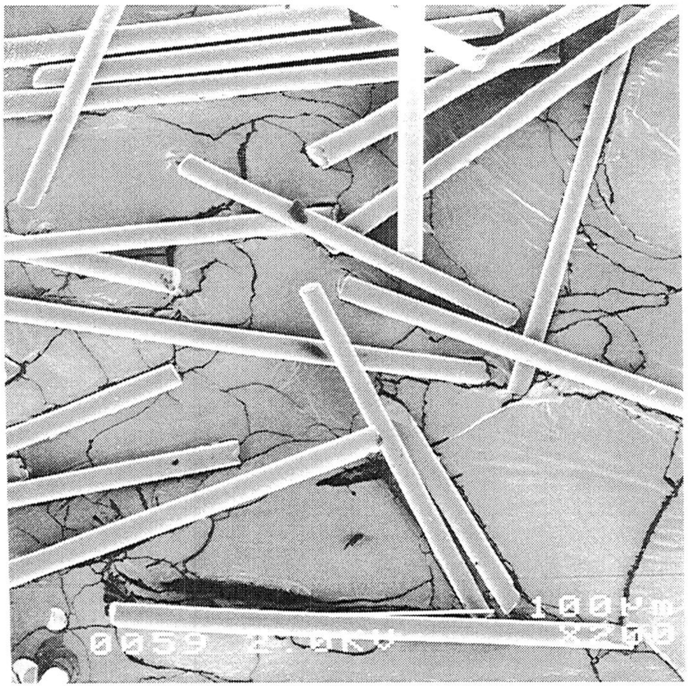


Fig. 42
Glass fibers 200X

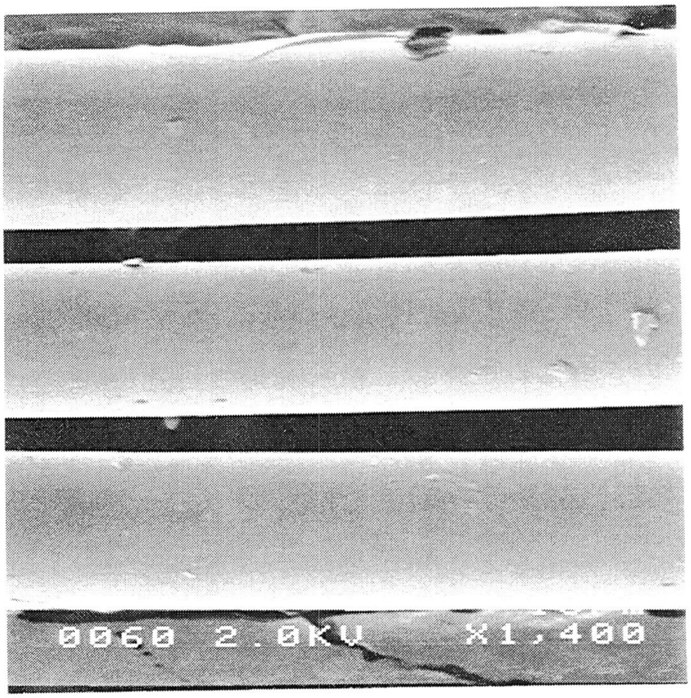


Fig. 43
Glass fiber surface
1,400X

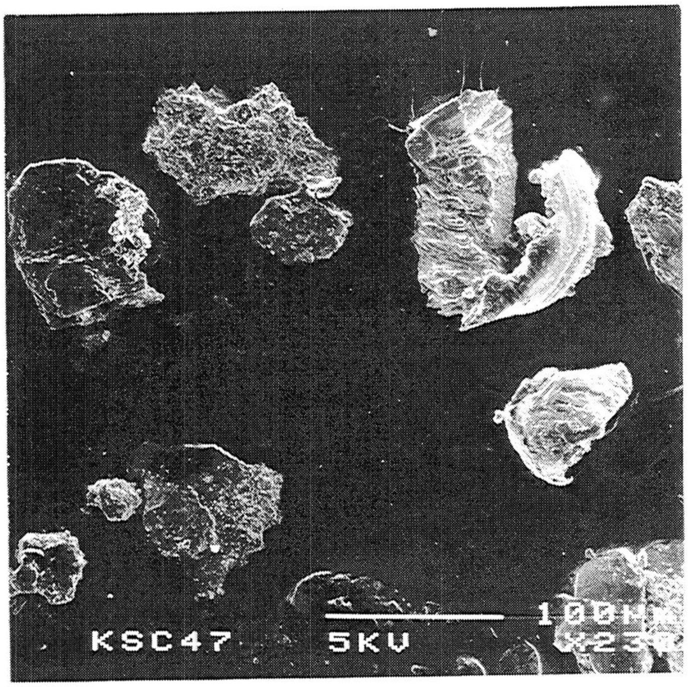


Fig. 44
Metallics 230X

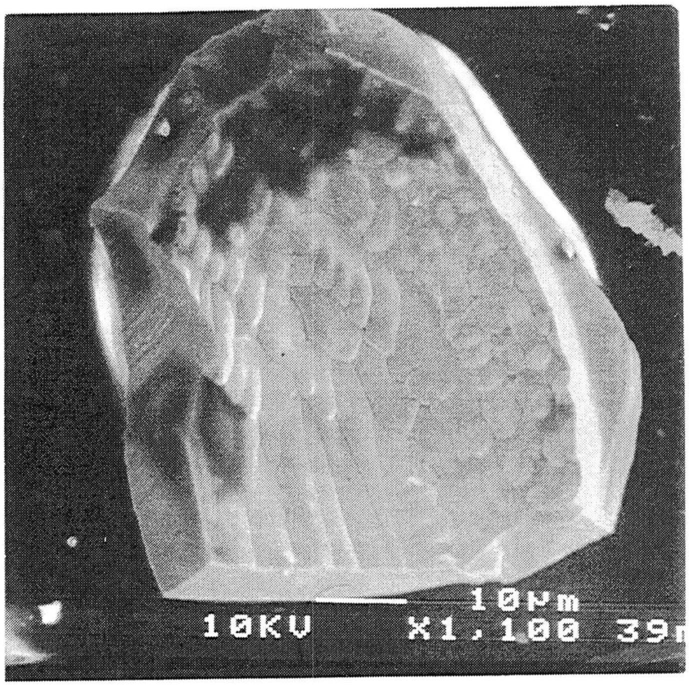


Fig. 45
Carbon steel 1,100X

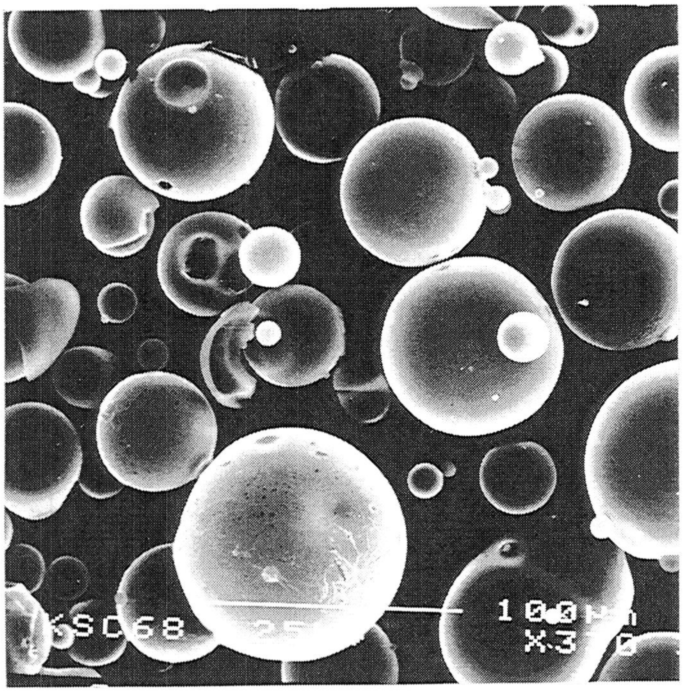


Fig. 46
Microballoons 330X

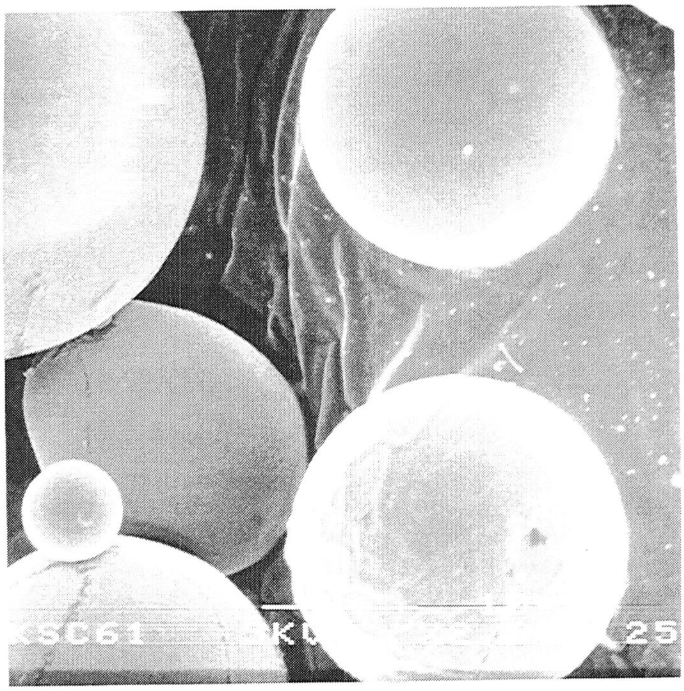


Fig. 47
Microballoons 2,000X

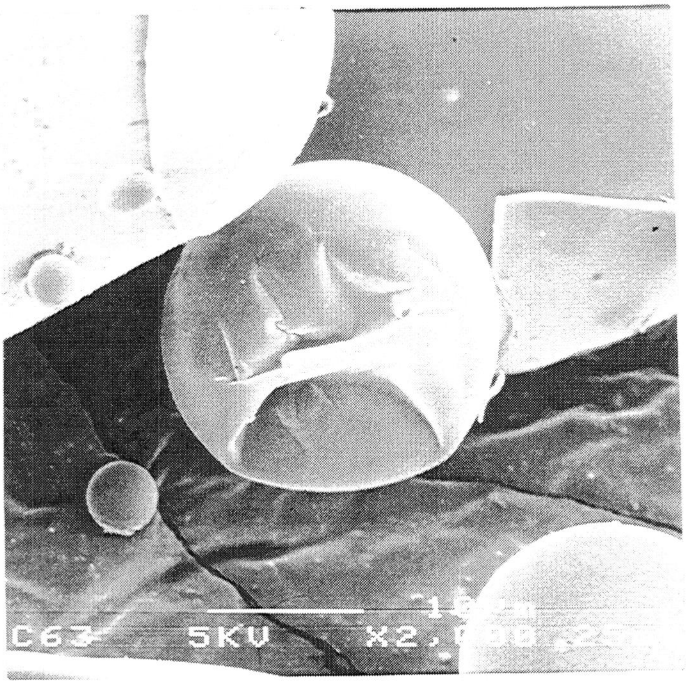


Fig. 48
Microballoons 2,000X

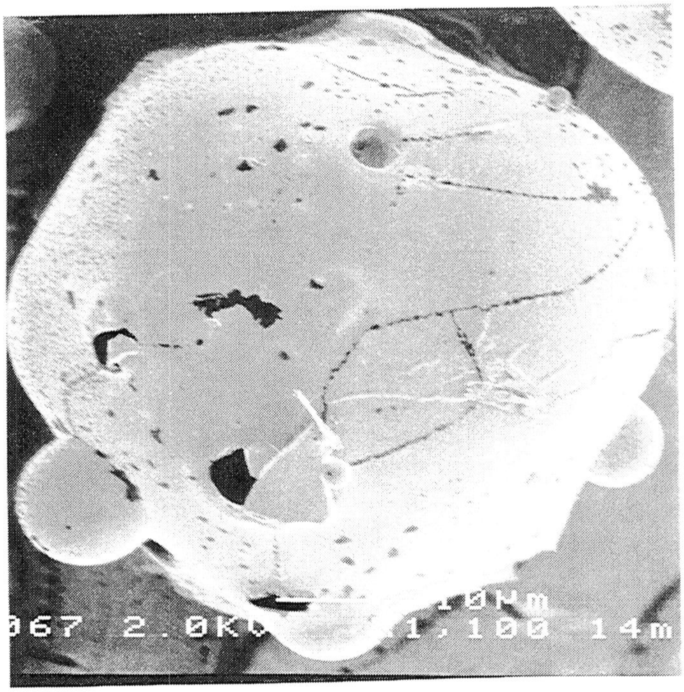


Fig. 49
Eccospheres 1,100X

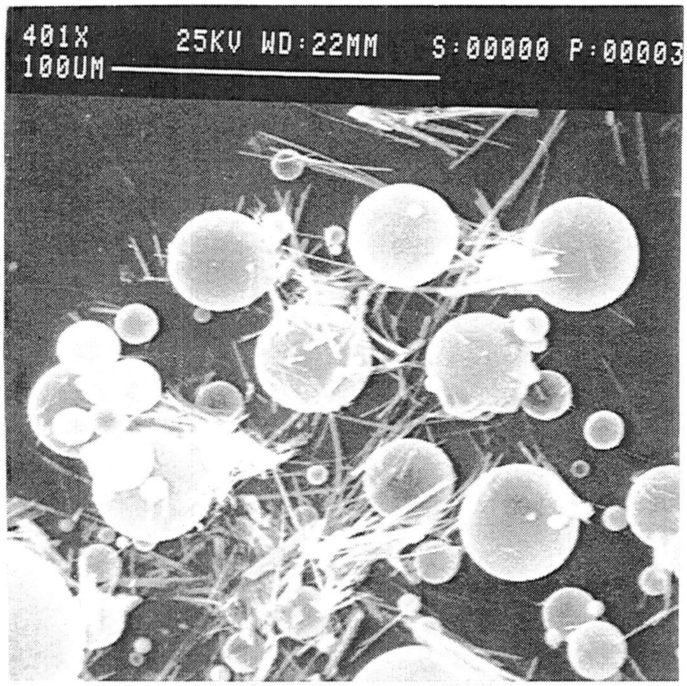


Fig. 50
Eccospheres 400X

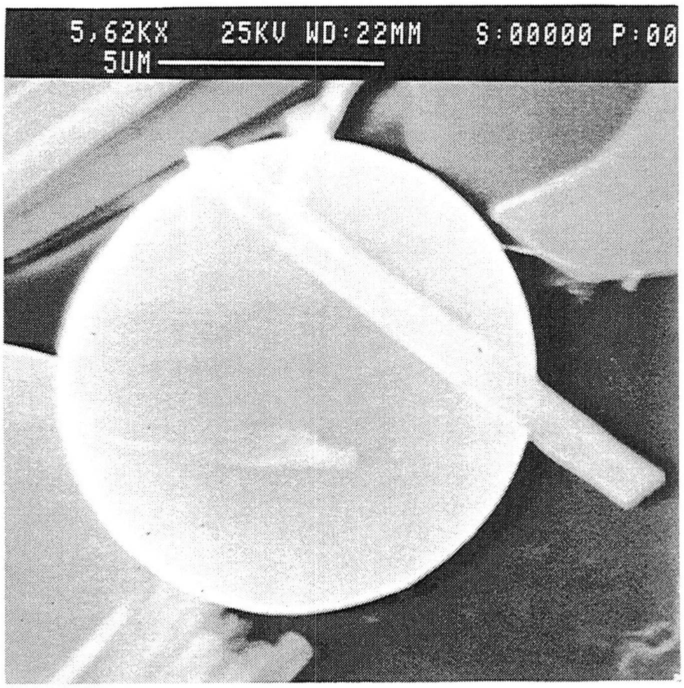


Fig. 51
Eccospheres 560X

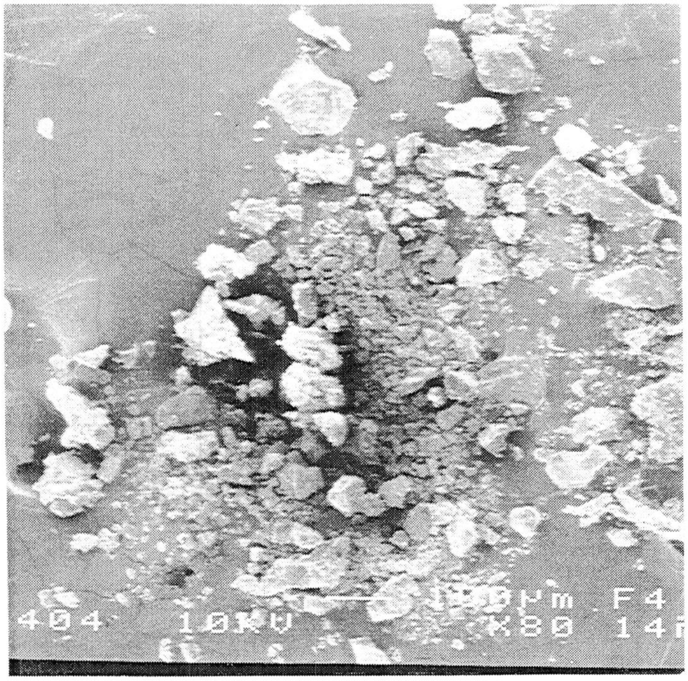


Fig. 52
MoS_2 particles 80X

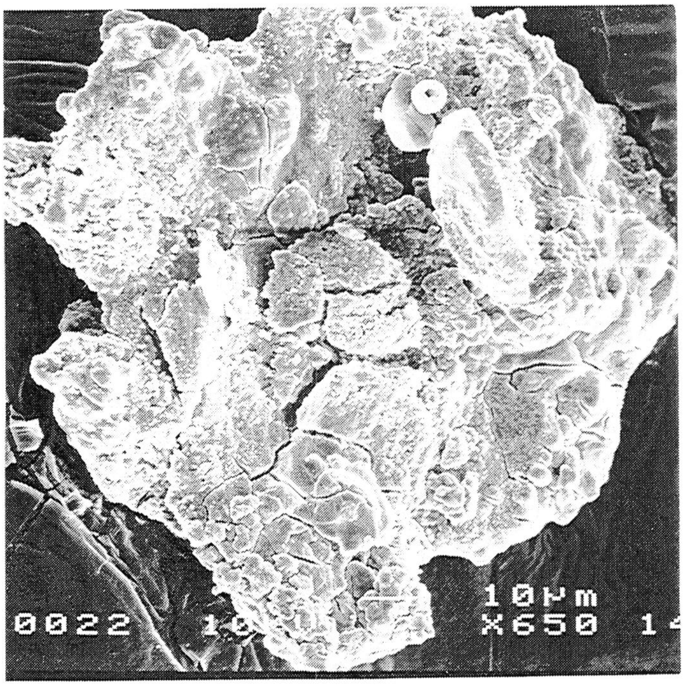


Fig. 53
MoS_2 650X

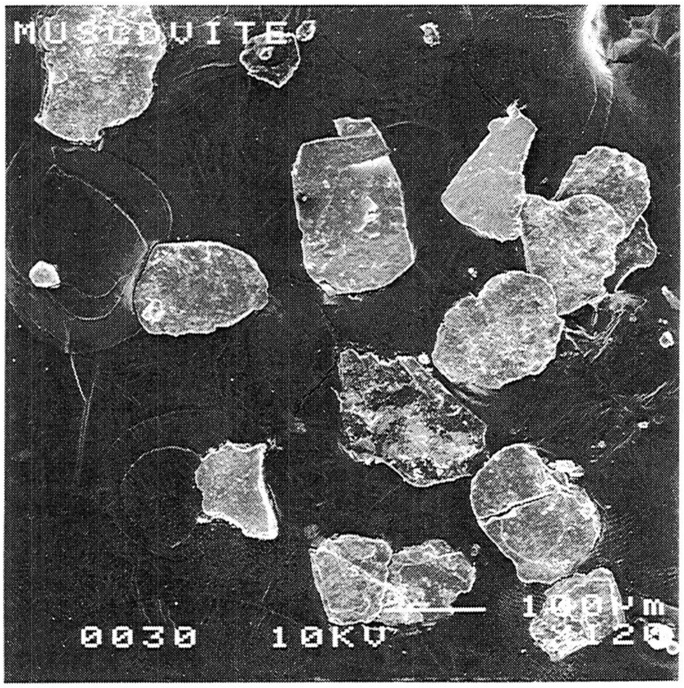


Fig. 54
Muscovite 120X

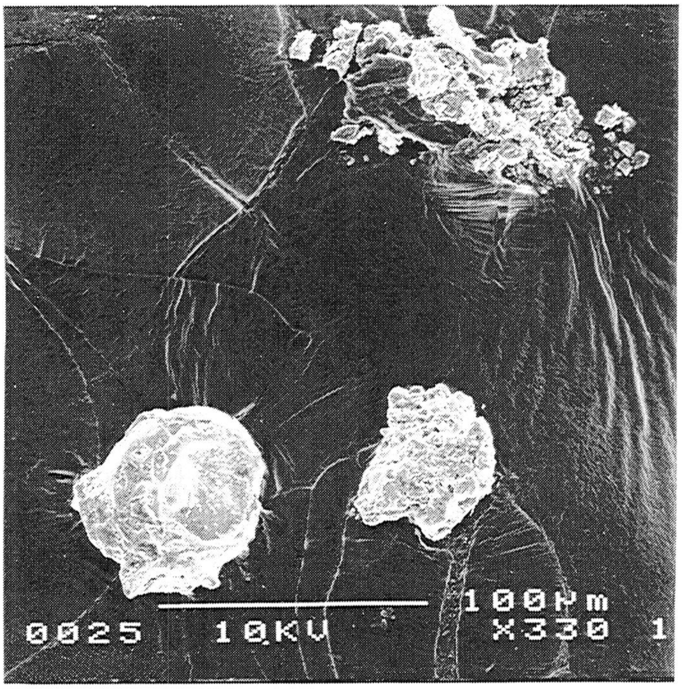


Fig. 55
Paint particles 330X

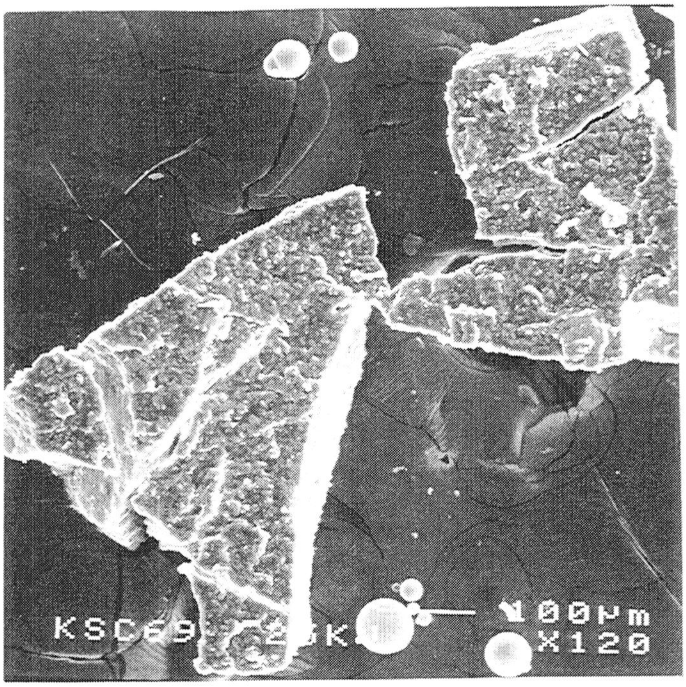


Fig. 56
RTV 120X

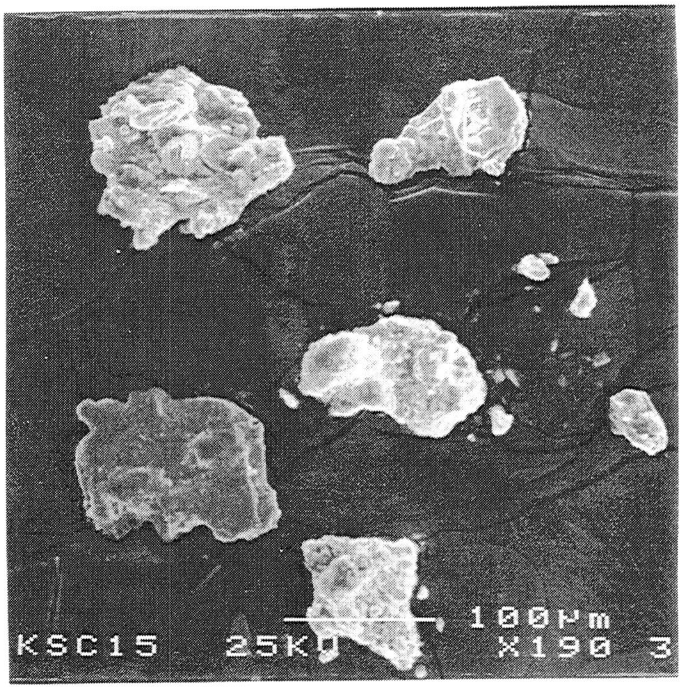


Fig. 57
Rust particles 190X

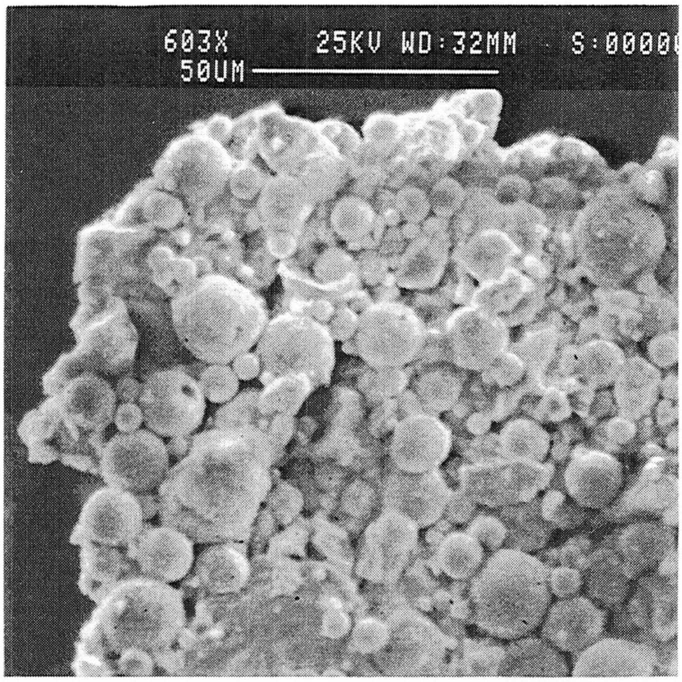


Fig. 58
Surfaces of rust particles
603X

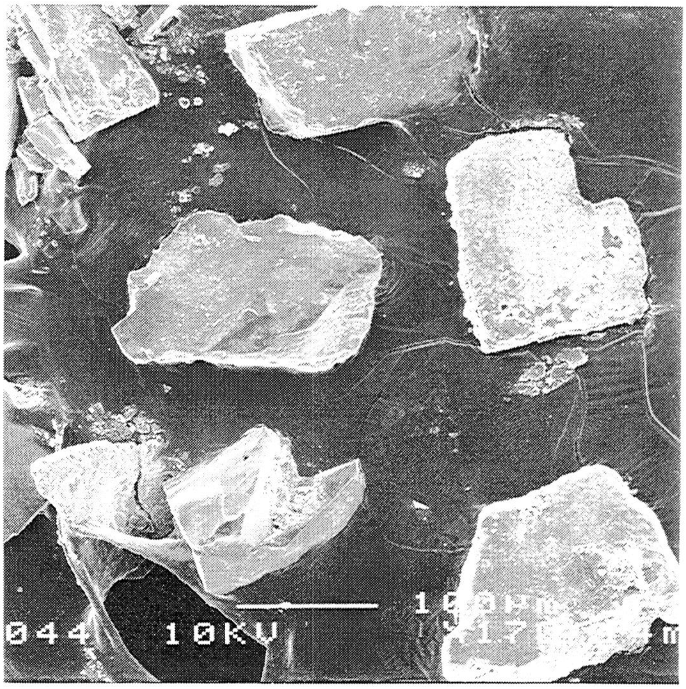


Fig. 59
Salt 170X

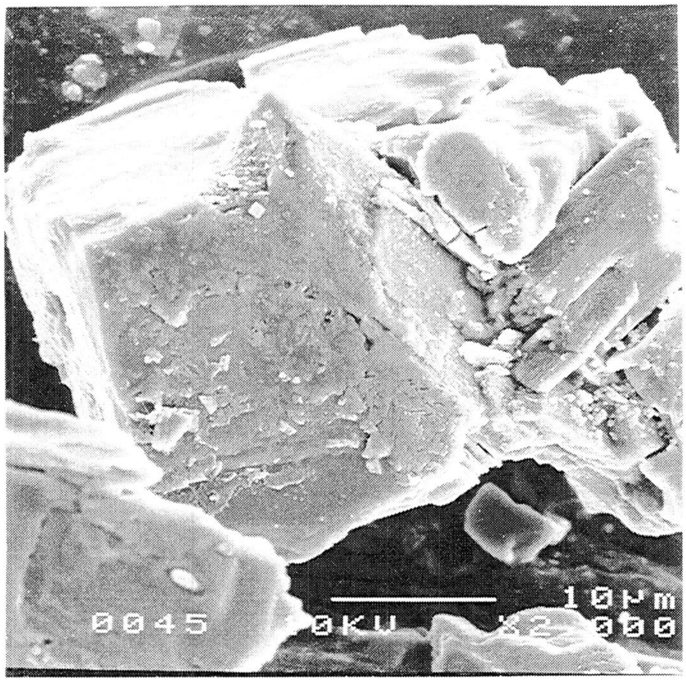


Fig. 60
Salt 2,000X

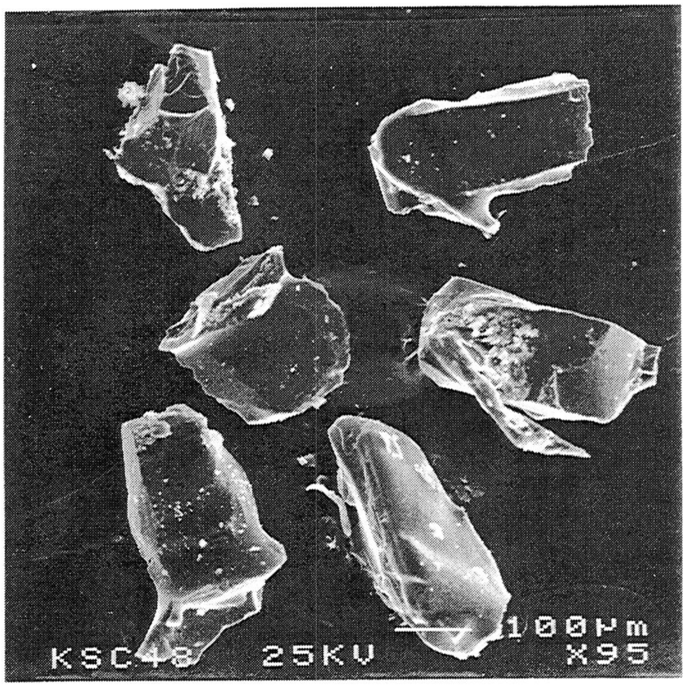


Fig. 61
Black SiC particles
95X

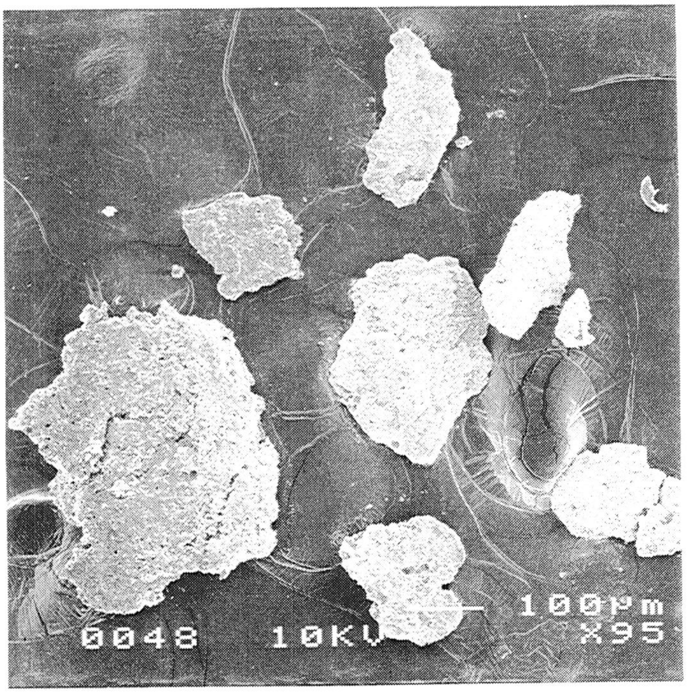


Fig. 62
White materials 95X

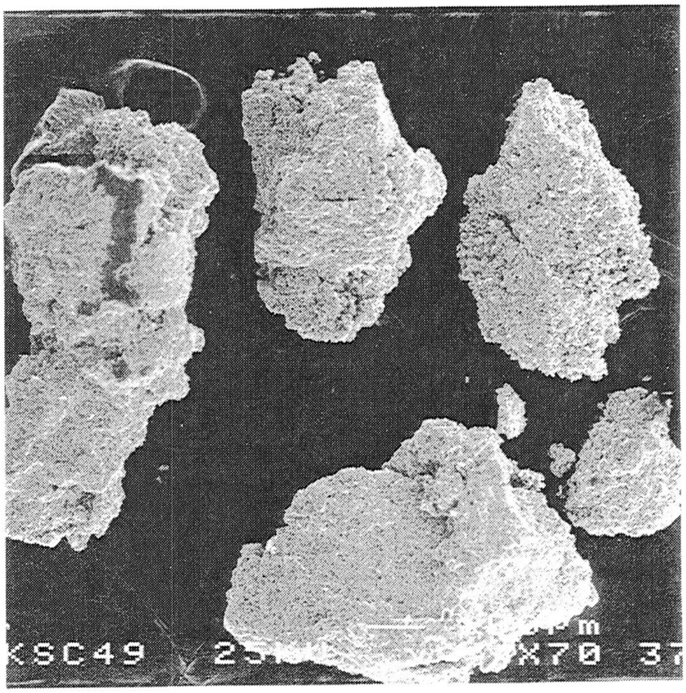


Fig. 63
Zinc primer 70X

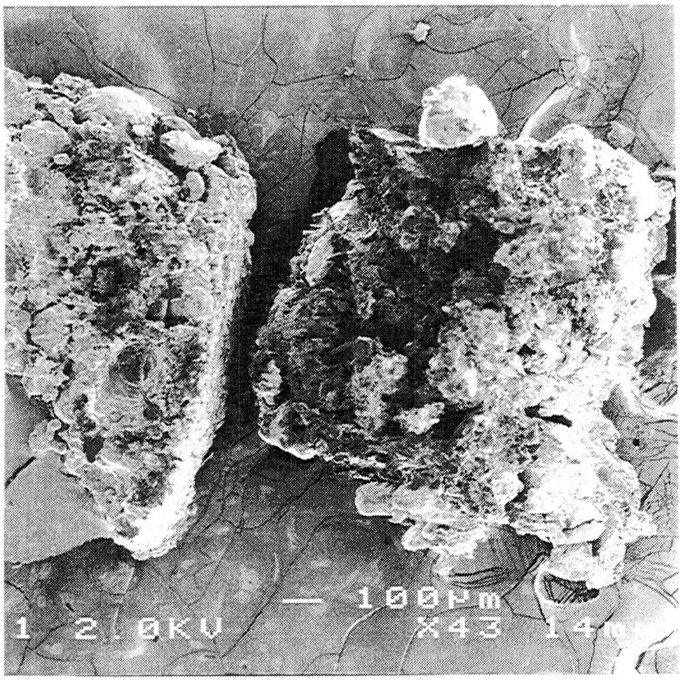


Fig. 64
Zinc primer 43X

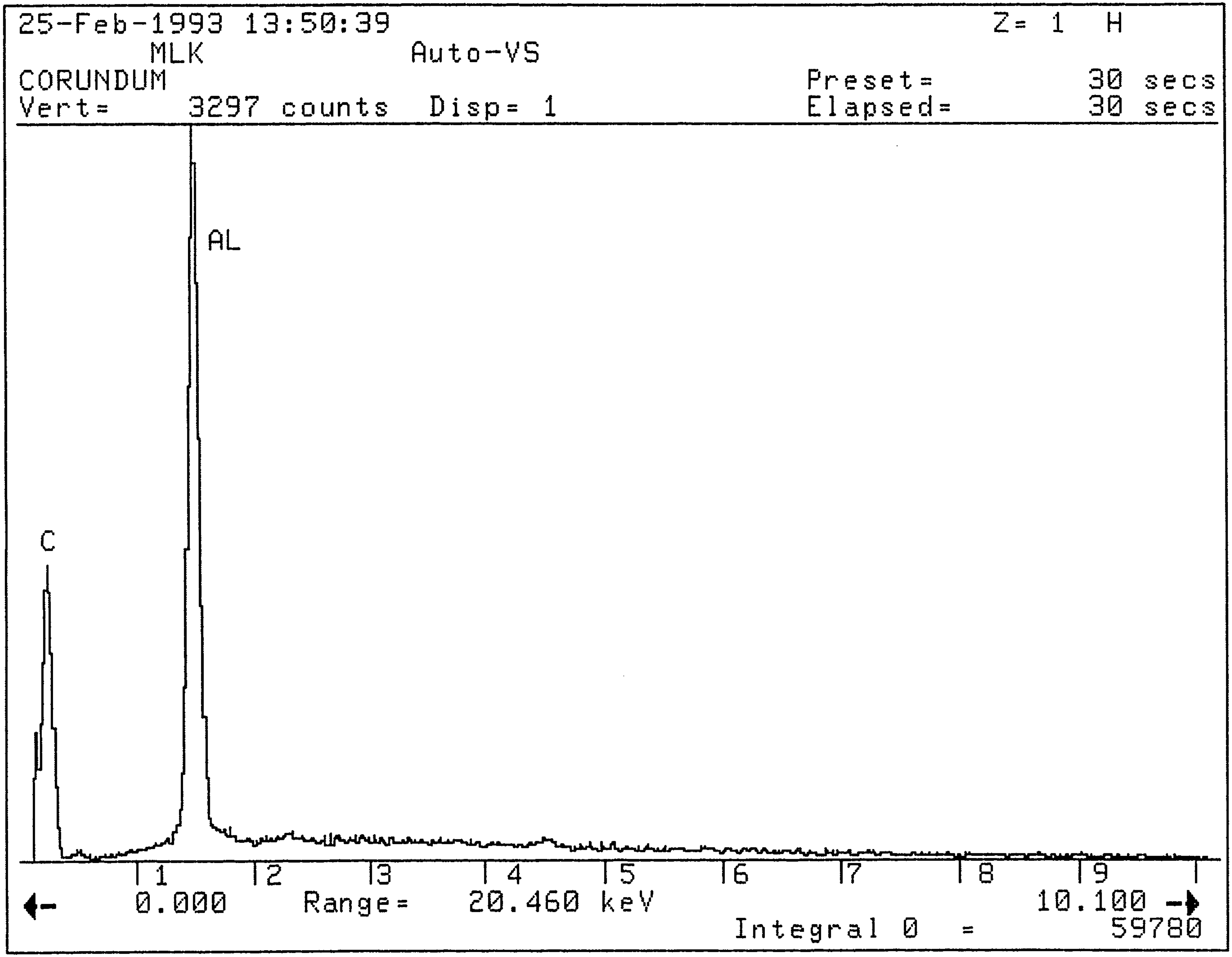


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Fig. 65 Energy Dispersive Spectrometry (EDS) spectrum of corundum

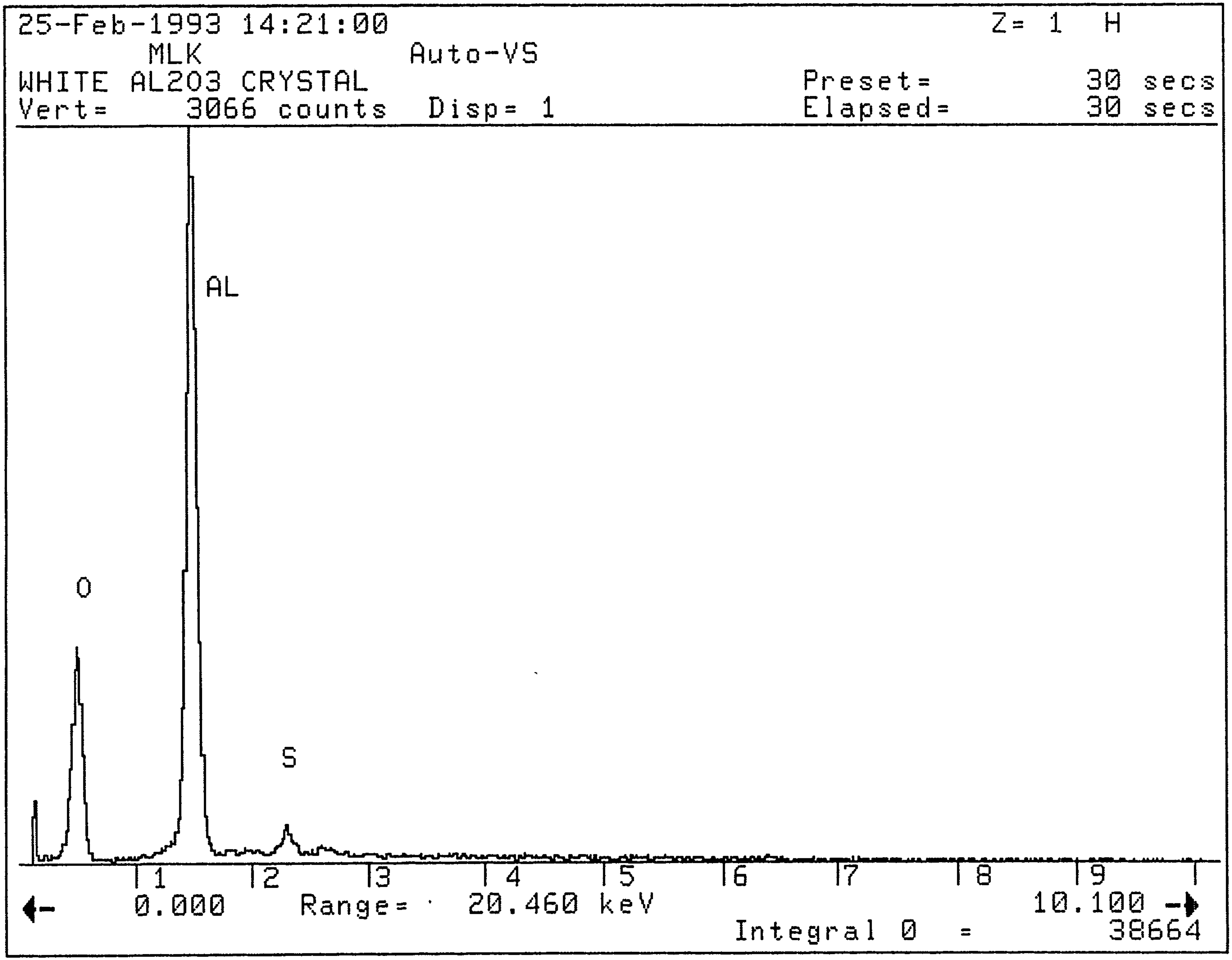

Fig. 66 EDS spectrum of white Al_2O_3 crystal

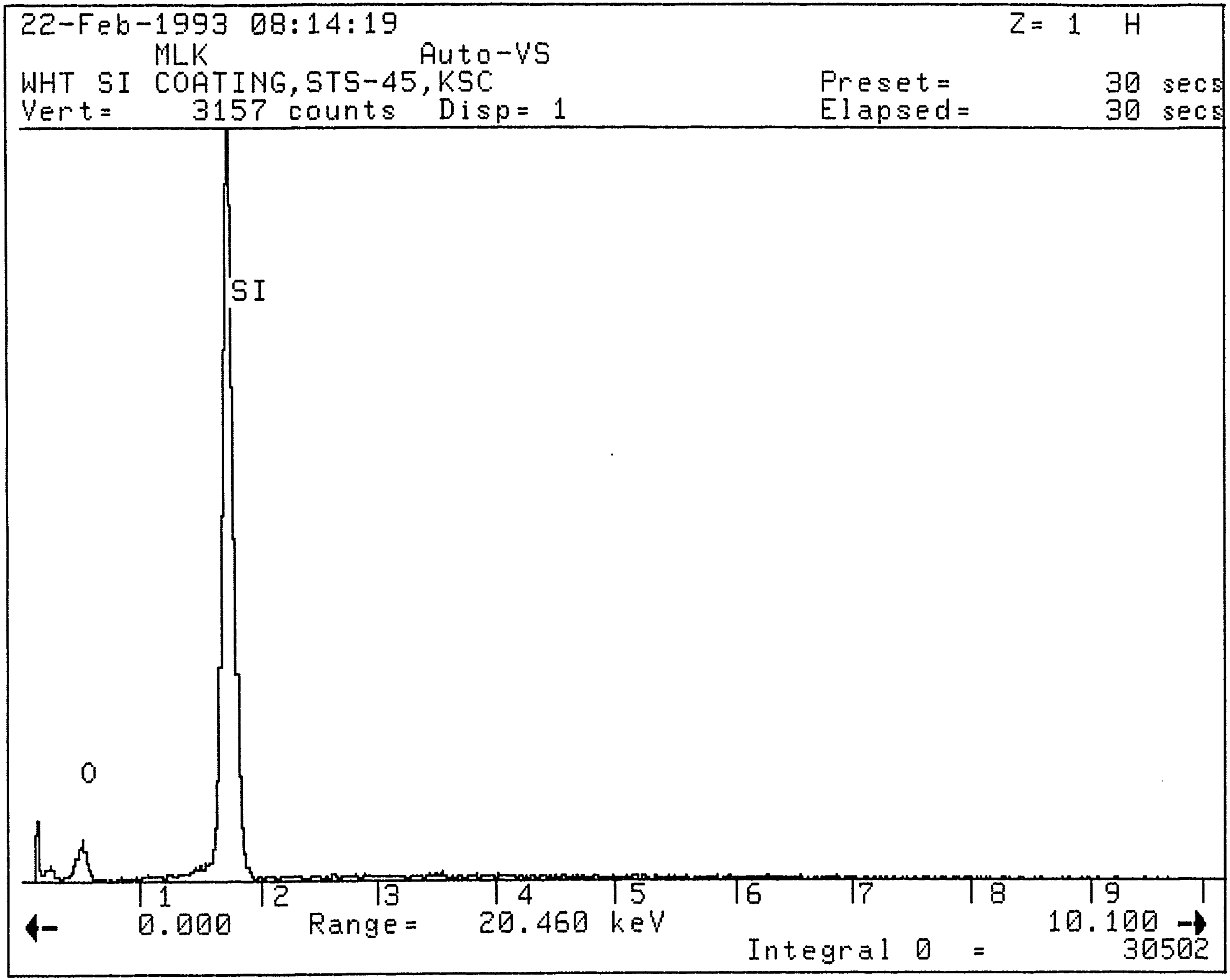

Fig. 67 EDS spectrum of white silica coating

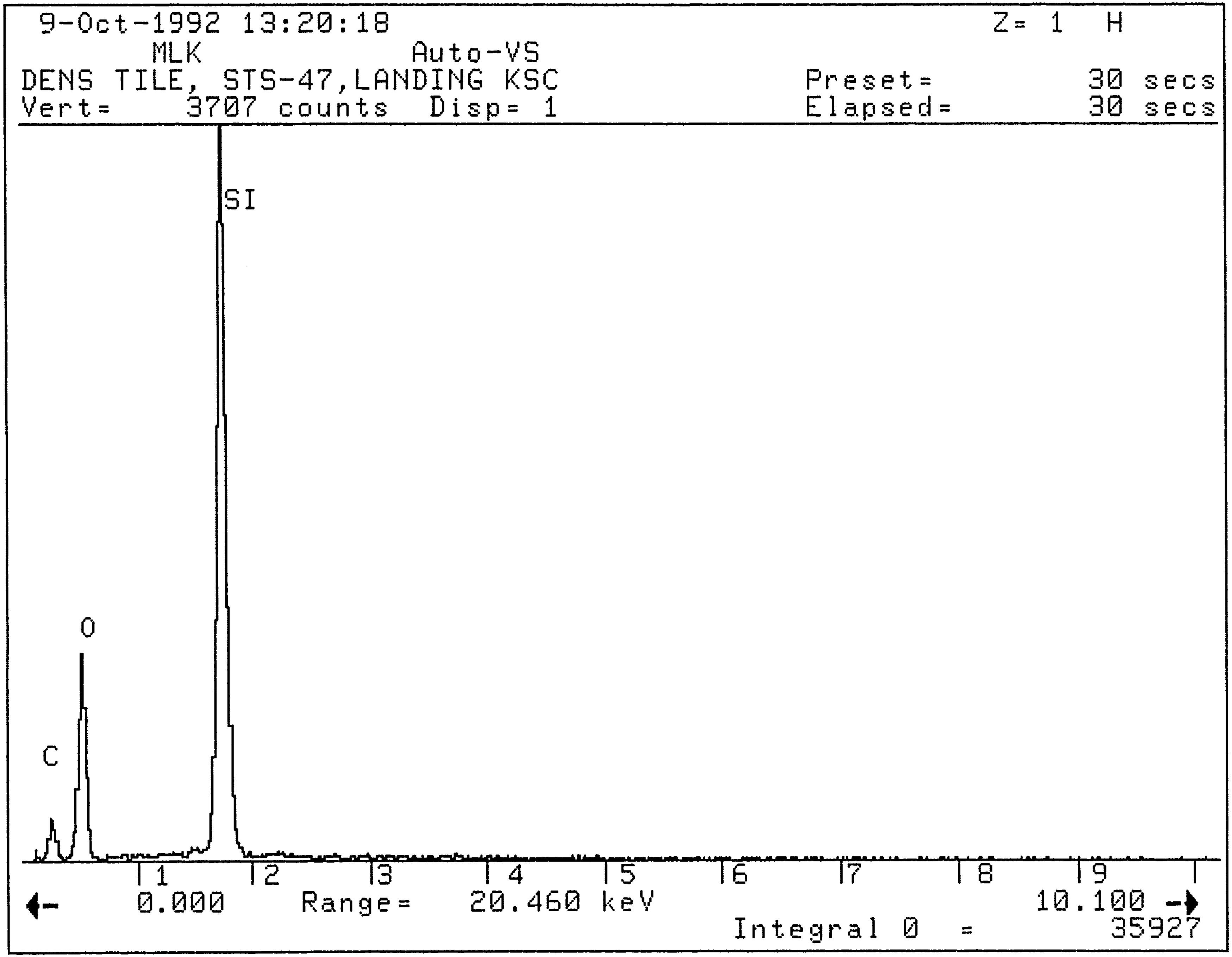

Fig. 68 EDS spectrum of dense tile

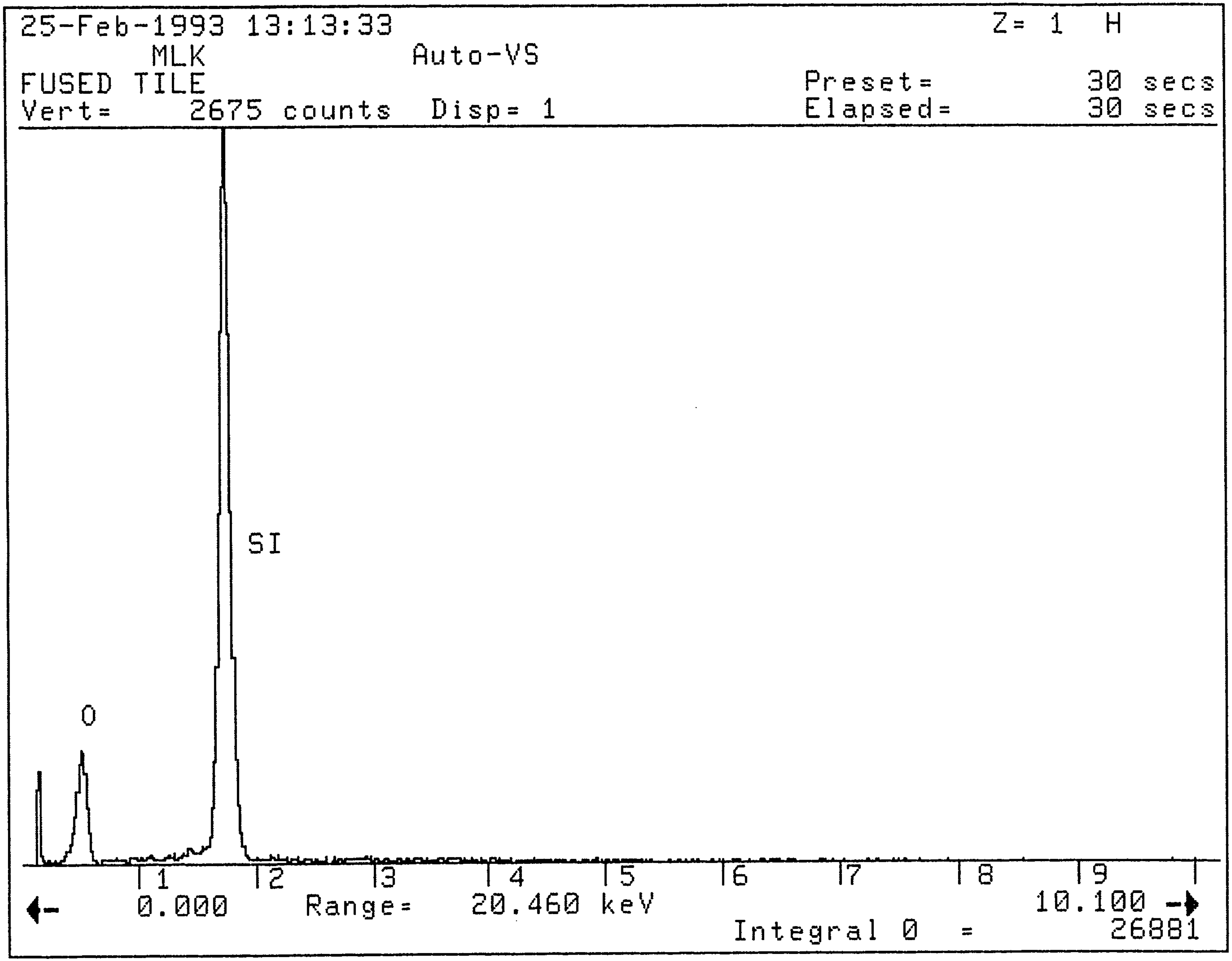

Fig. 69 EDS spectrum of fused tile

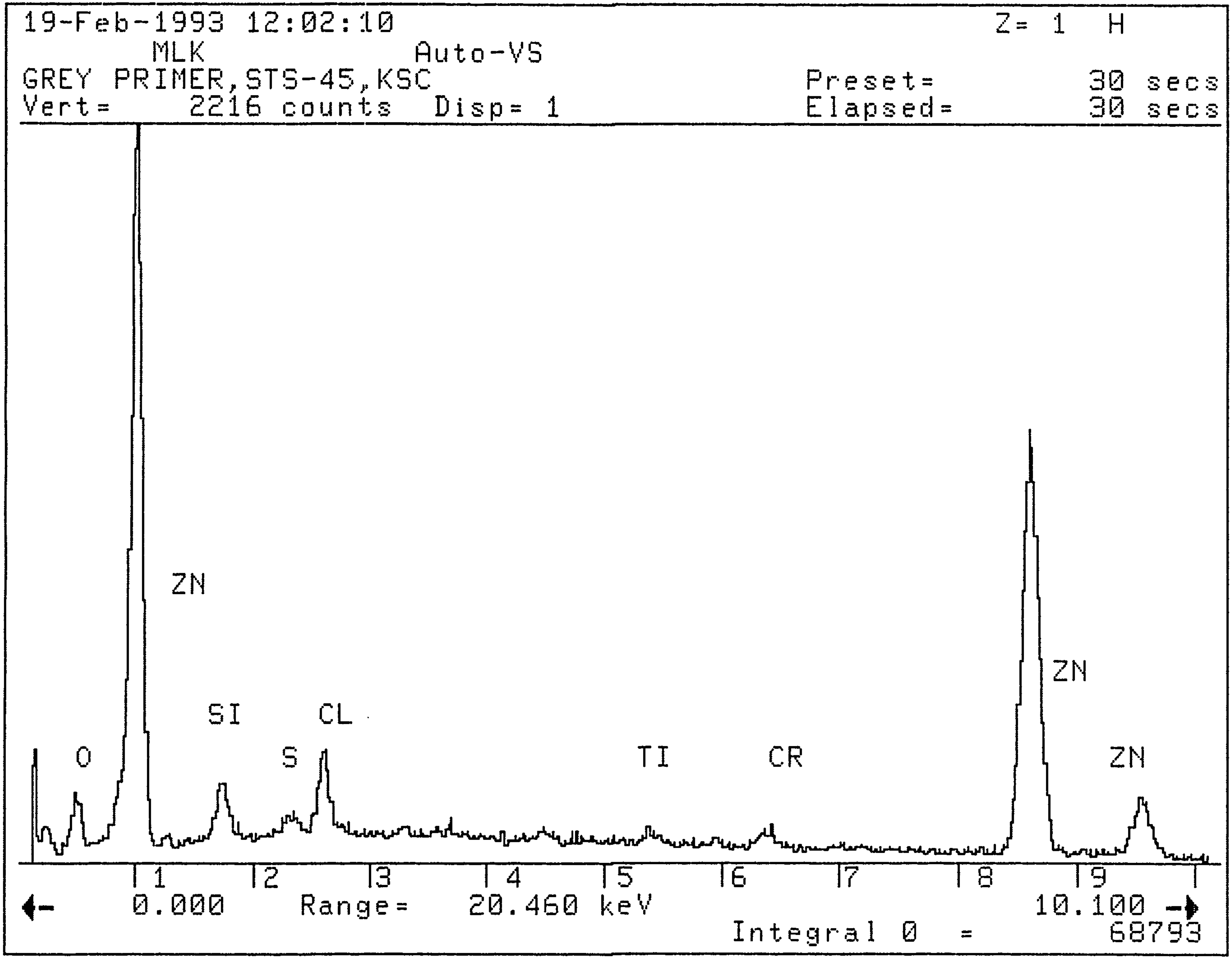

Fig. 70 EDS spectrum of light grey primer

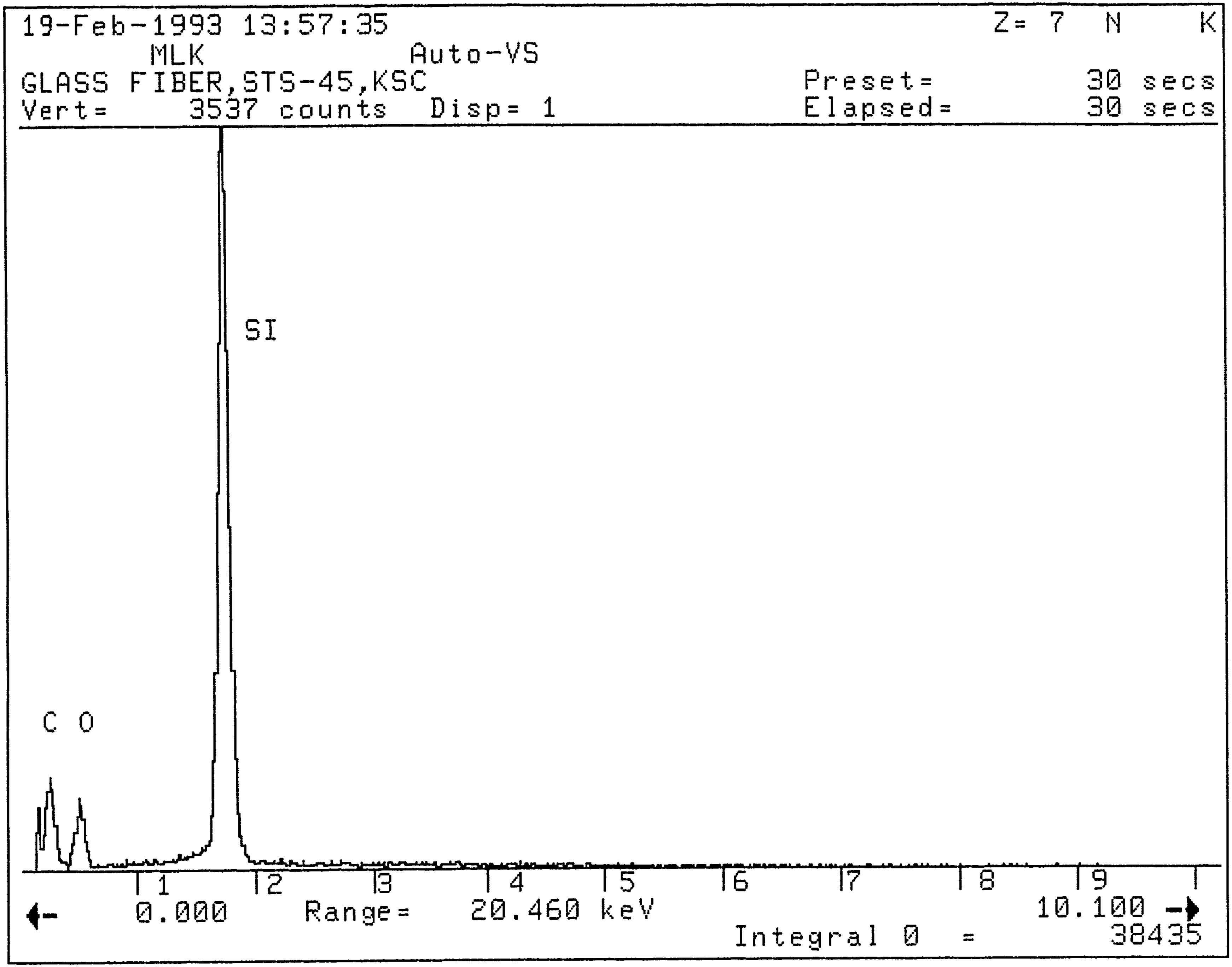

Fig. 71 EDS spectrum of tile glass fiber

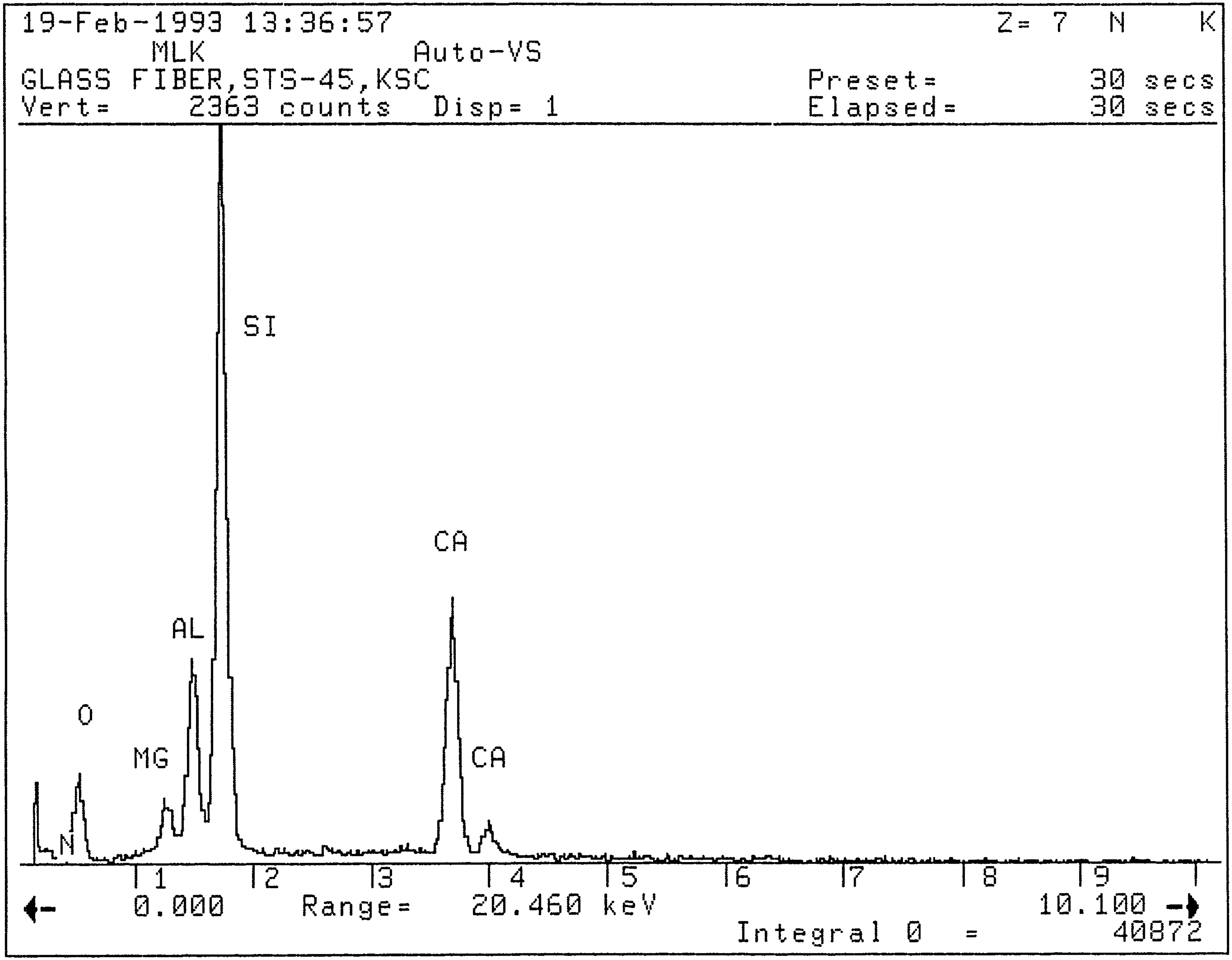

Fig. 72 EDS spectrum of insulation glass fiber

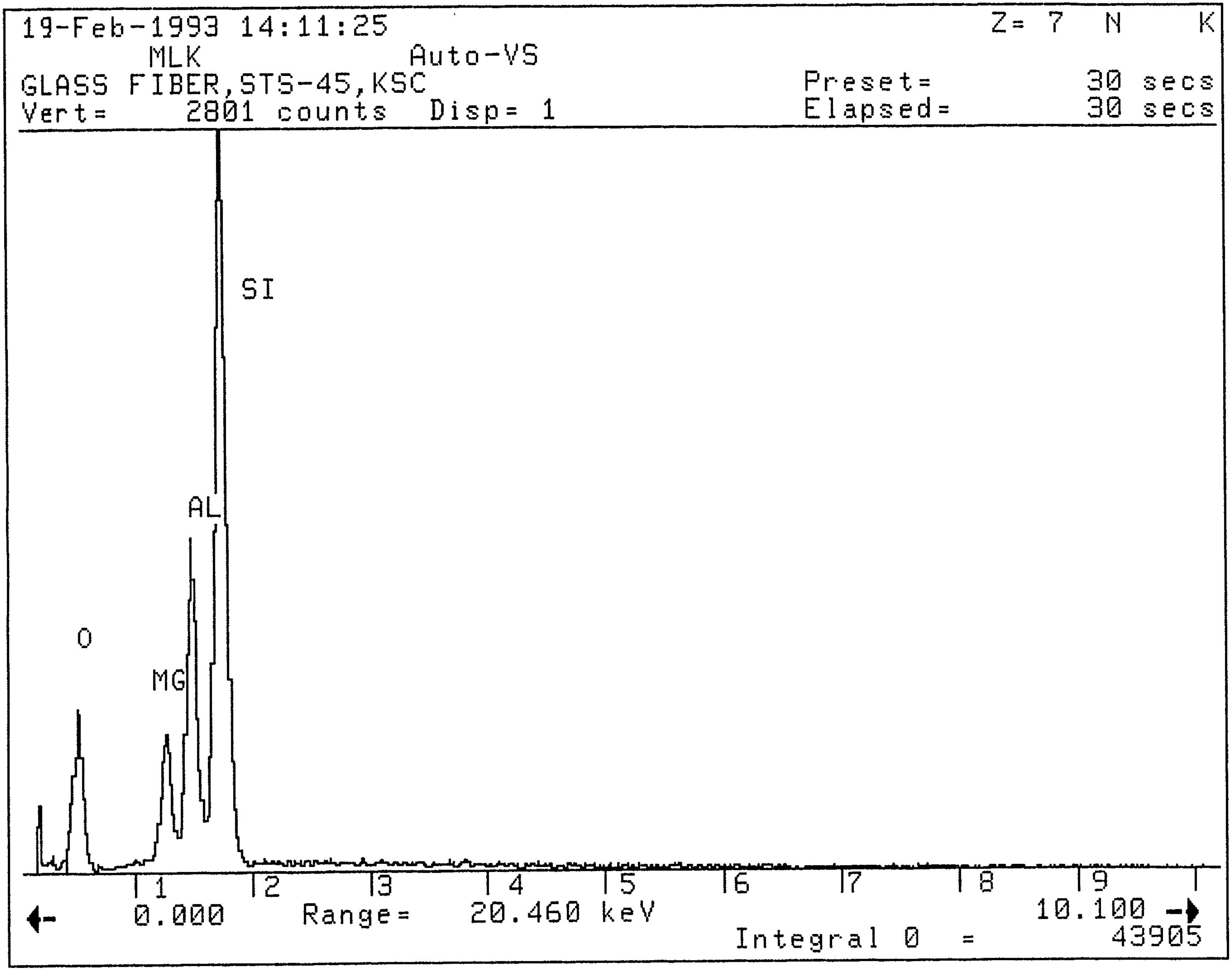

Fig. 73 EDS spectrum of high temperature insulation glass fiber

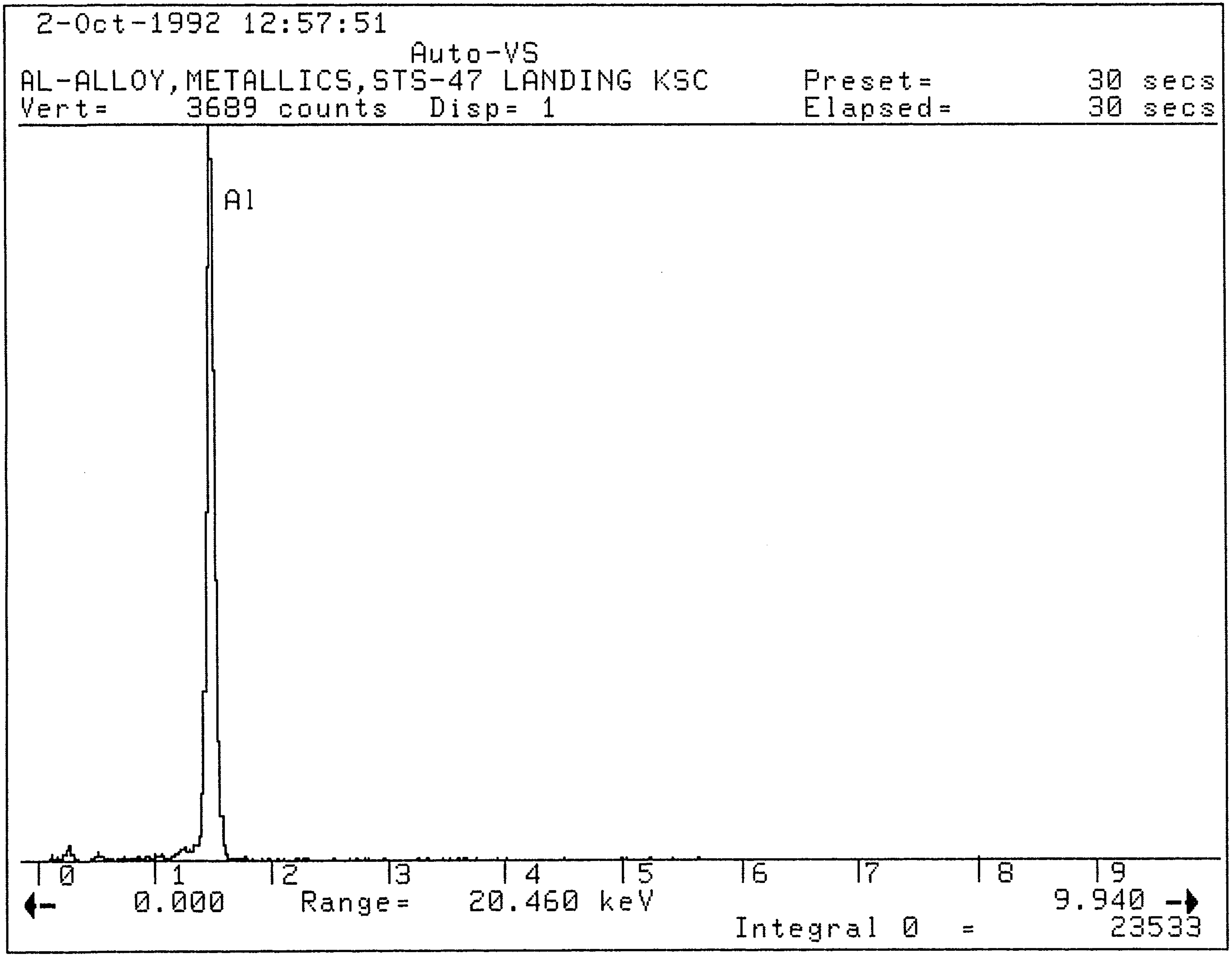

Fig. 74 EDS spectrum of Al alloy

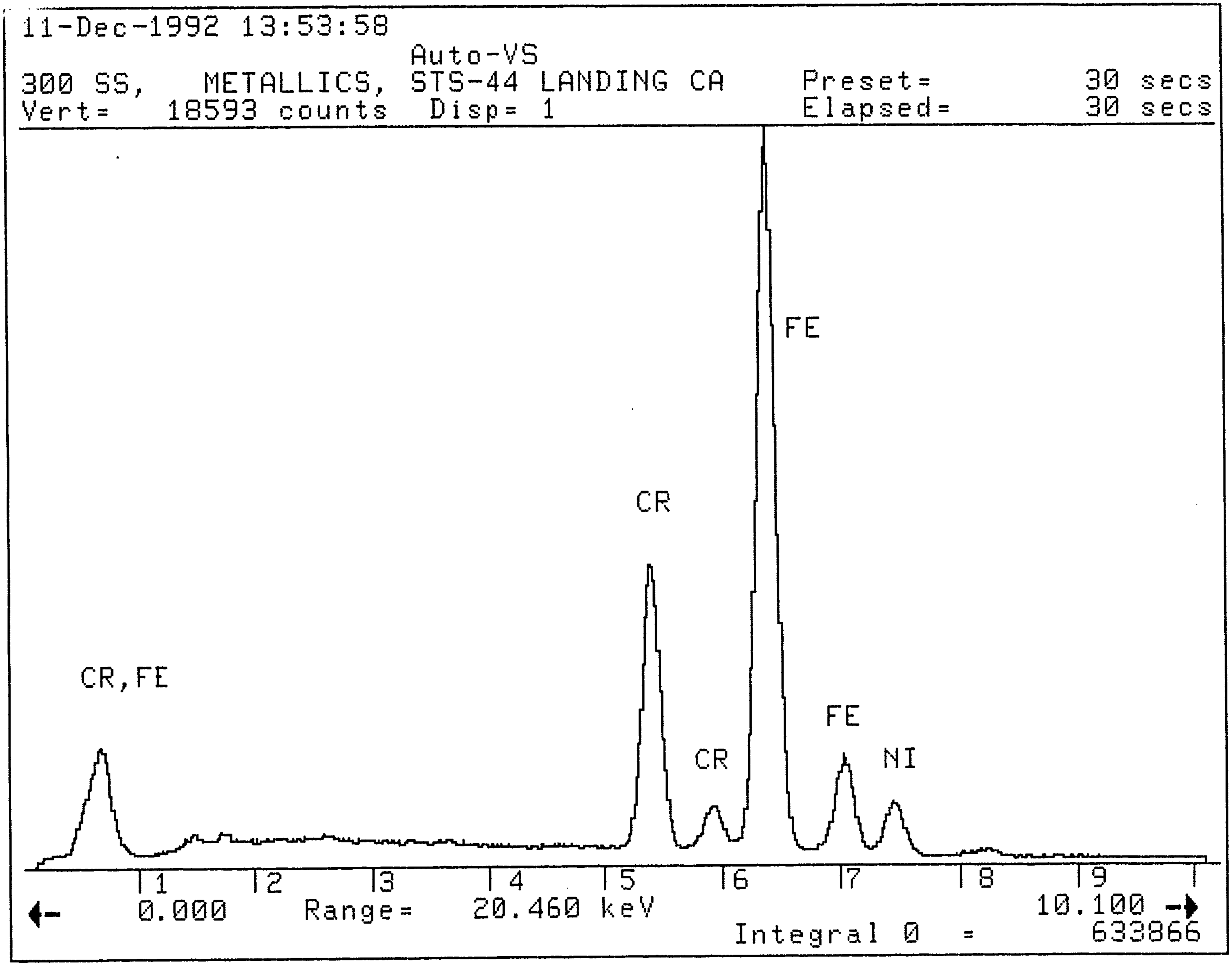

Fig. 75 EDS spectrum of a 300 series stainless steel

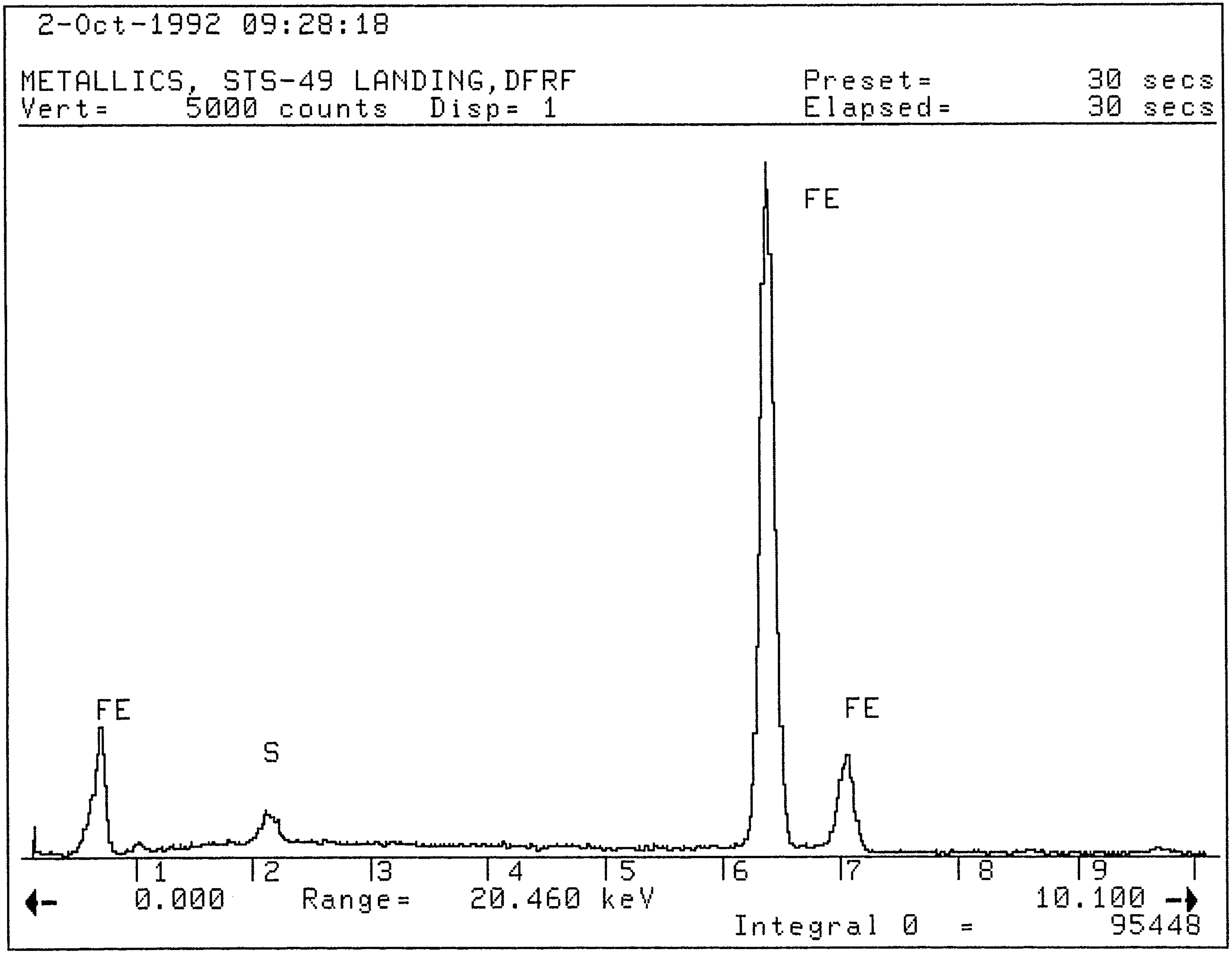

Fig. 76 EDS spectrum of carbon steel

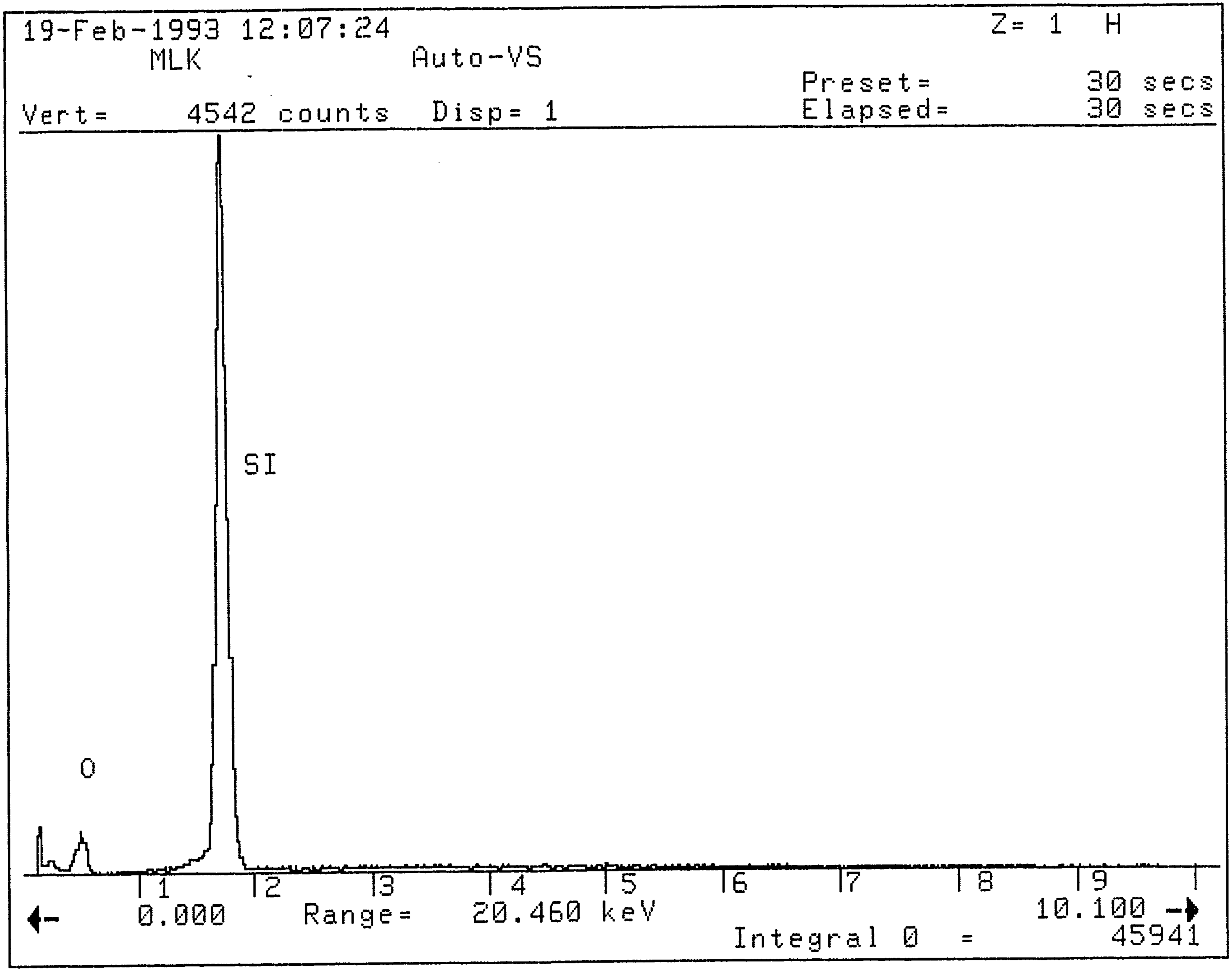

Fig. 77 EDS spectrum of eccosphere

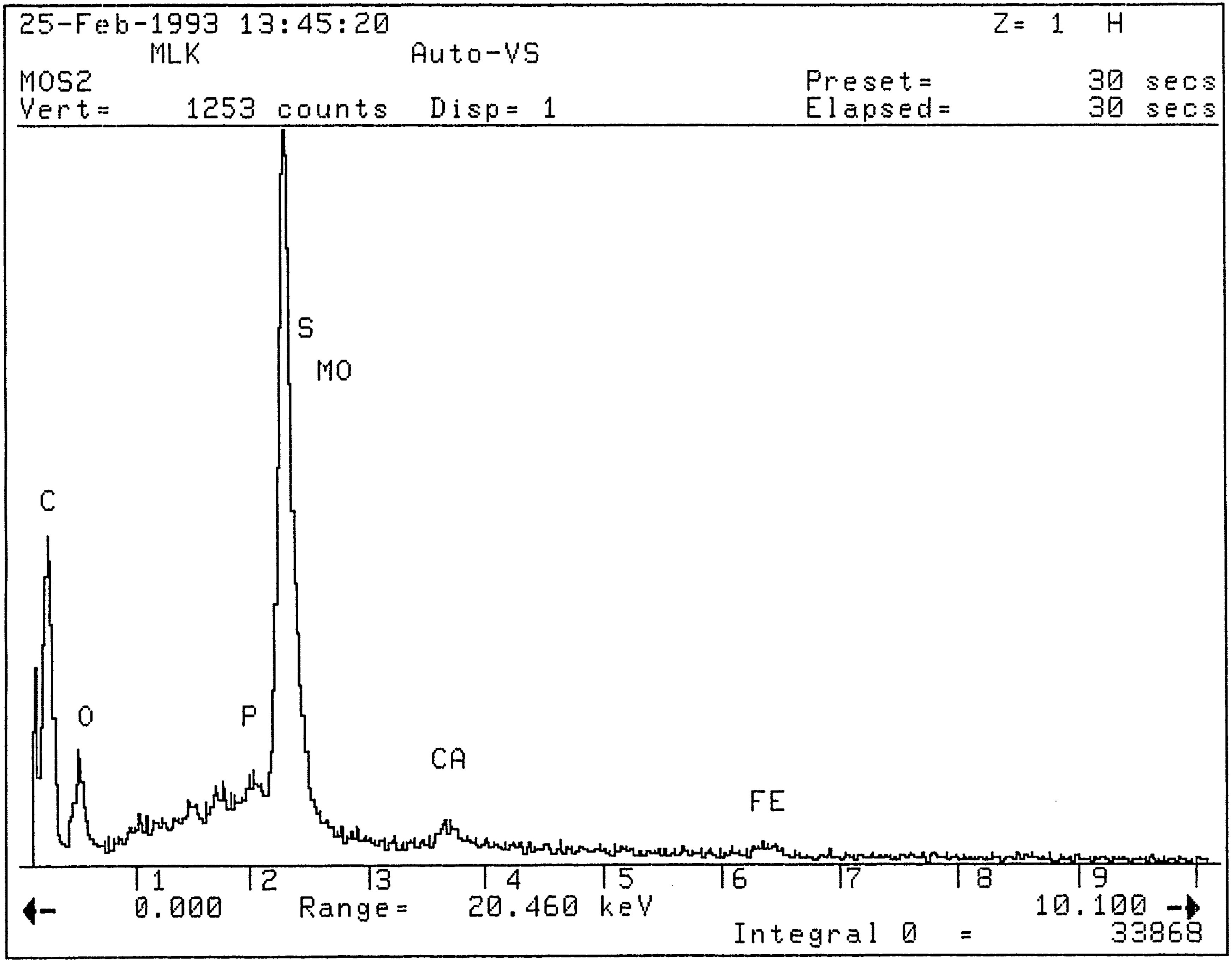

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Fig. 78 EDS spectrum of MoS_2

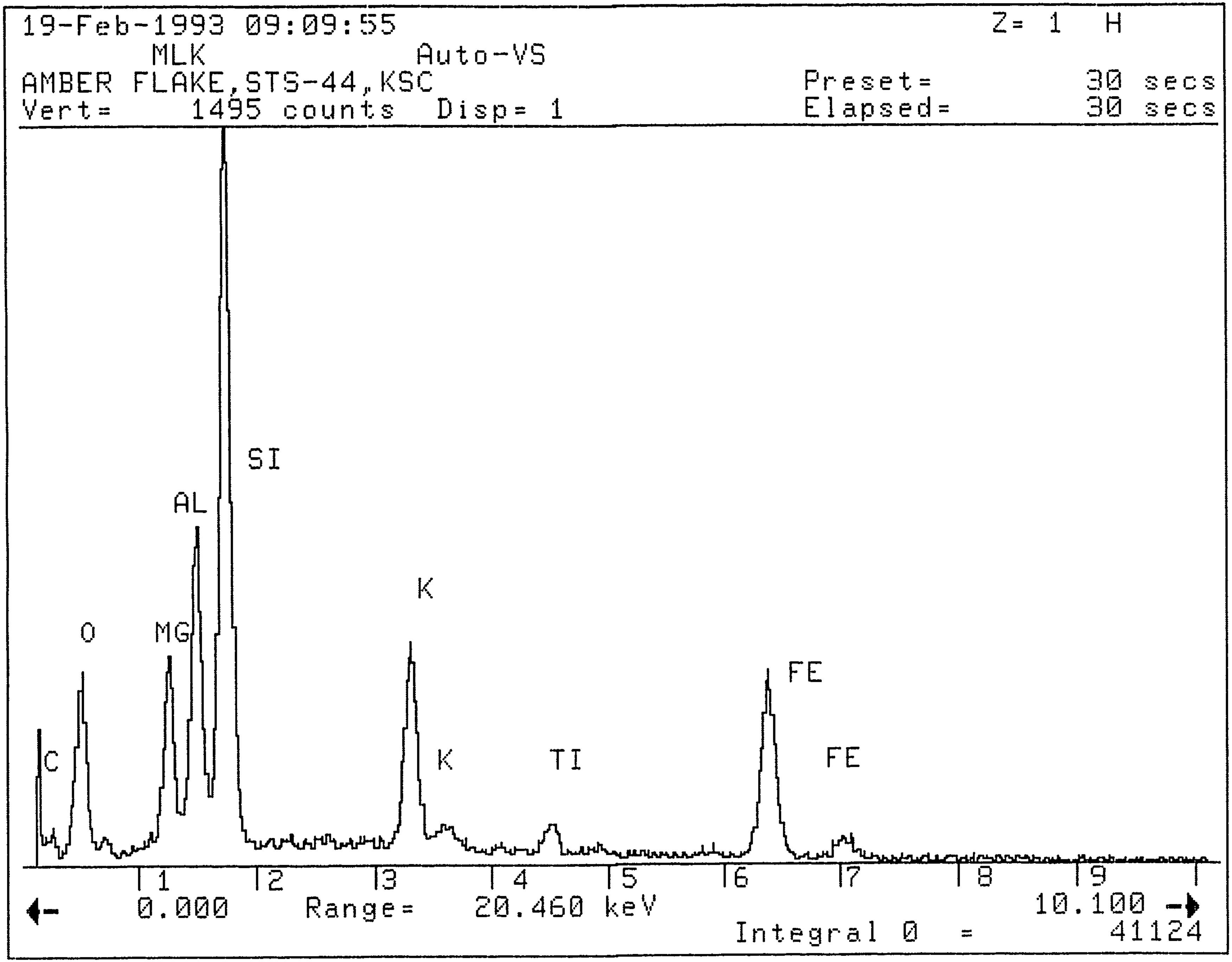

Fig. 79 EDS spectrum of amber flake (muscovite)

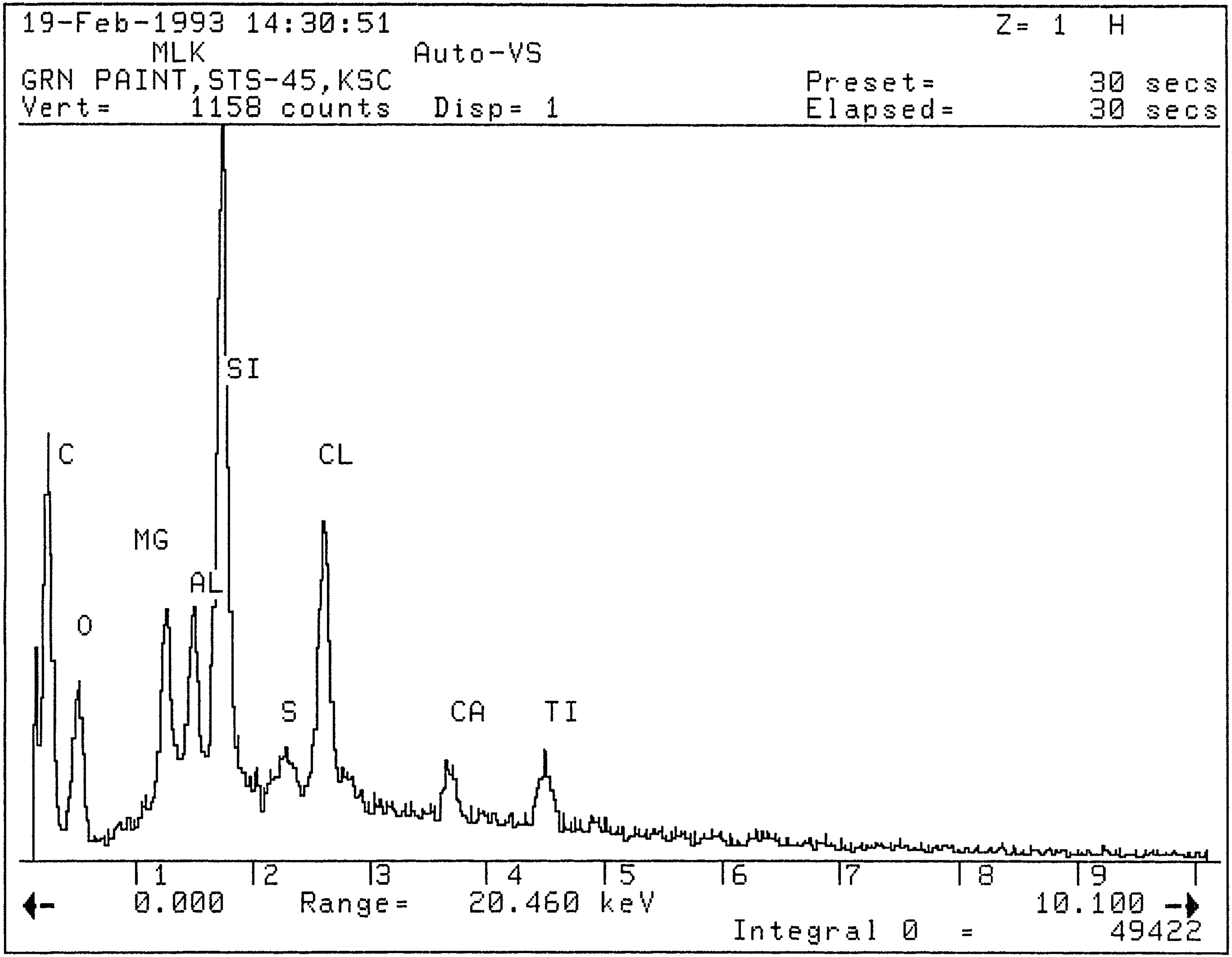

Fig. 80 EDS spectrum of green paint

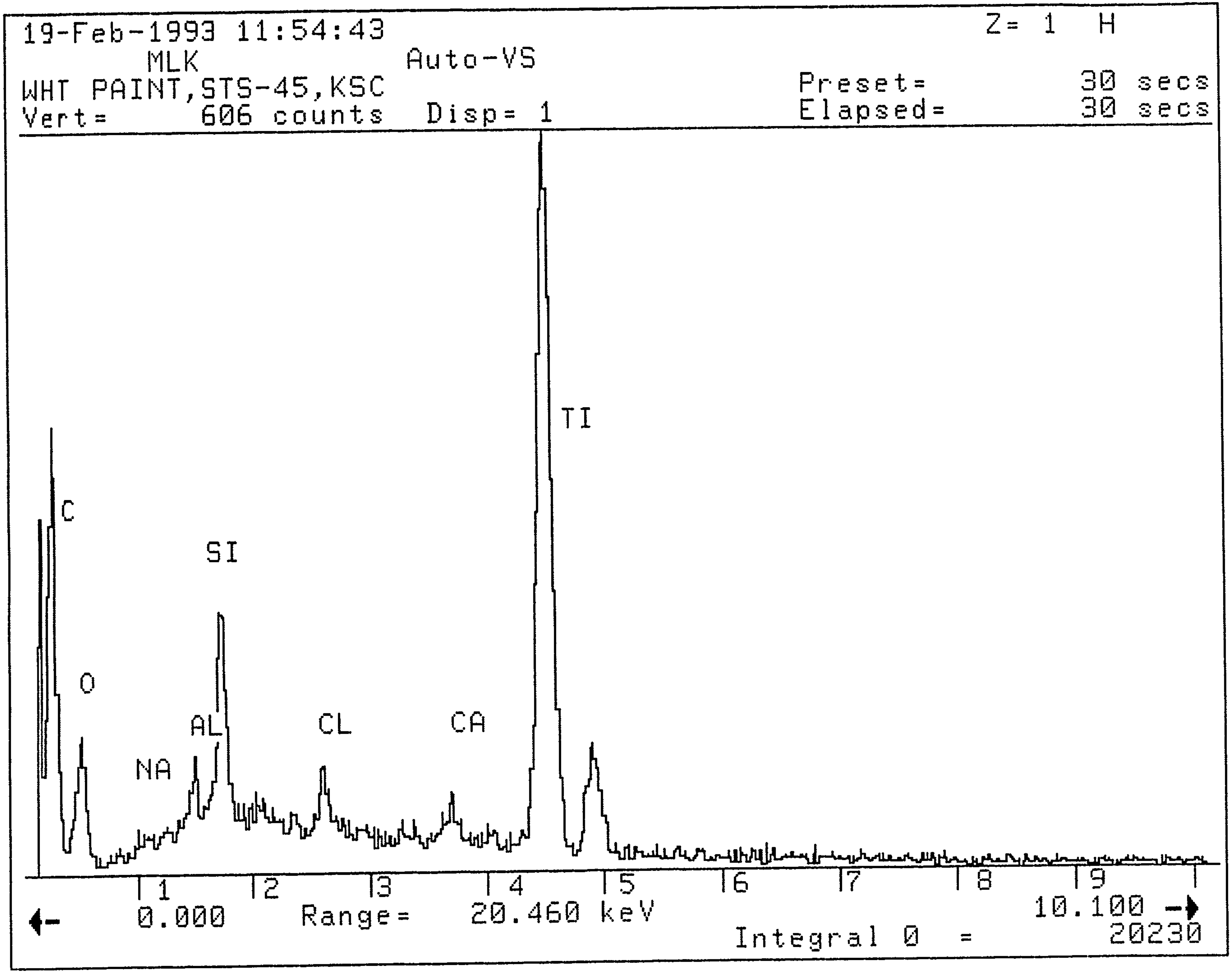

Fig. 81 EDS spectrum of white paint

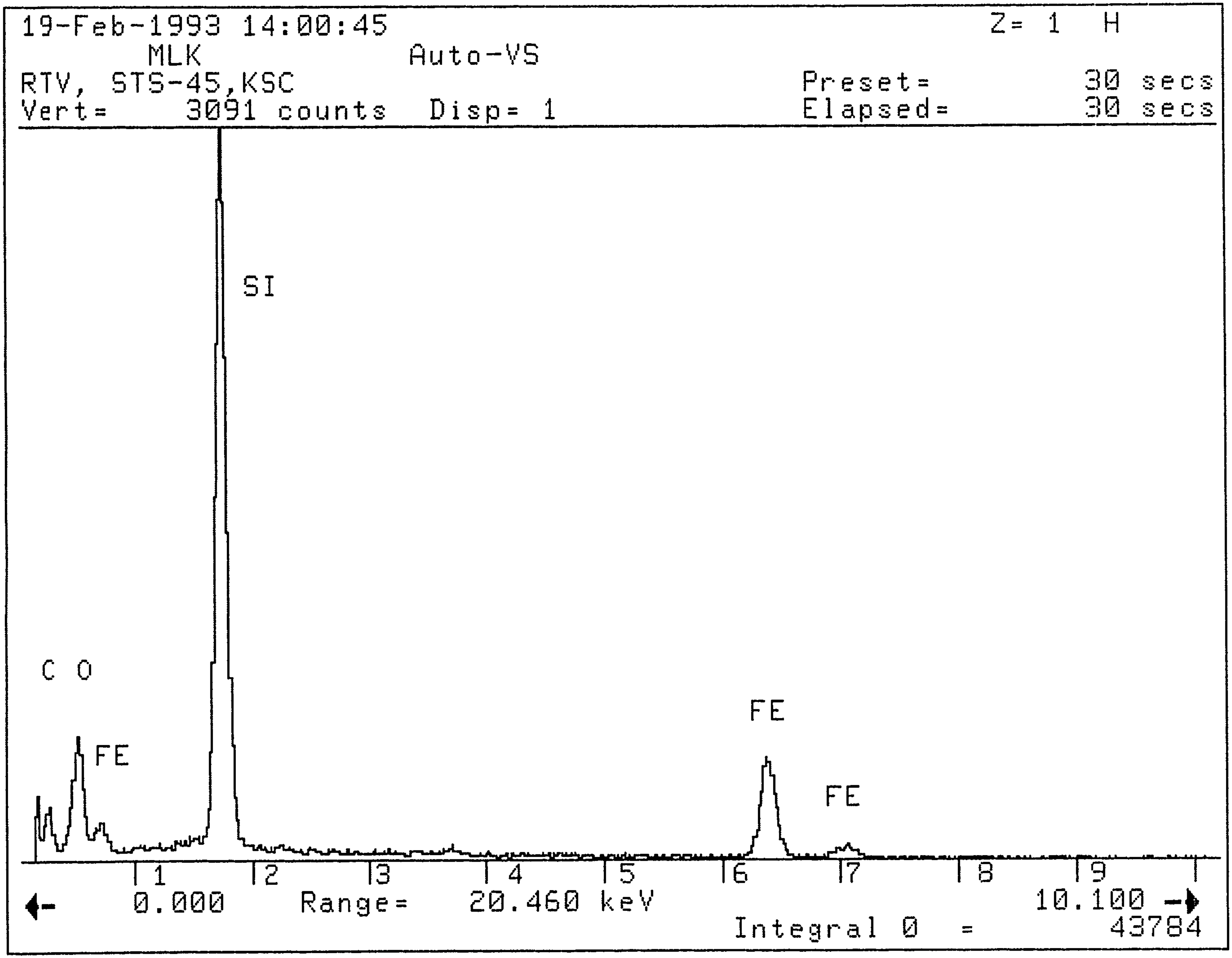

Fig. 82 EDS spectrum of RTV

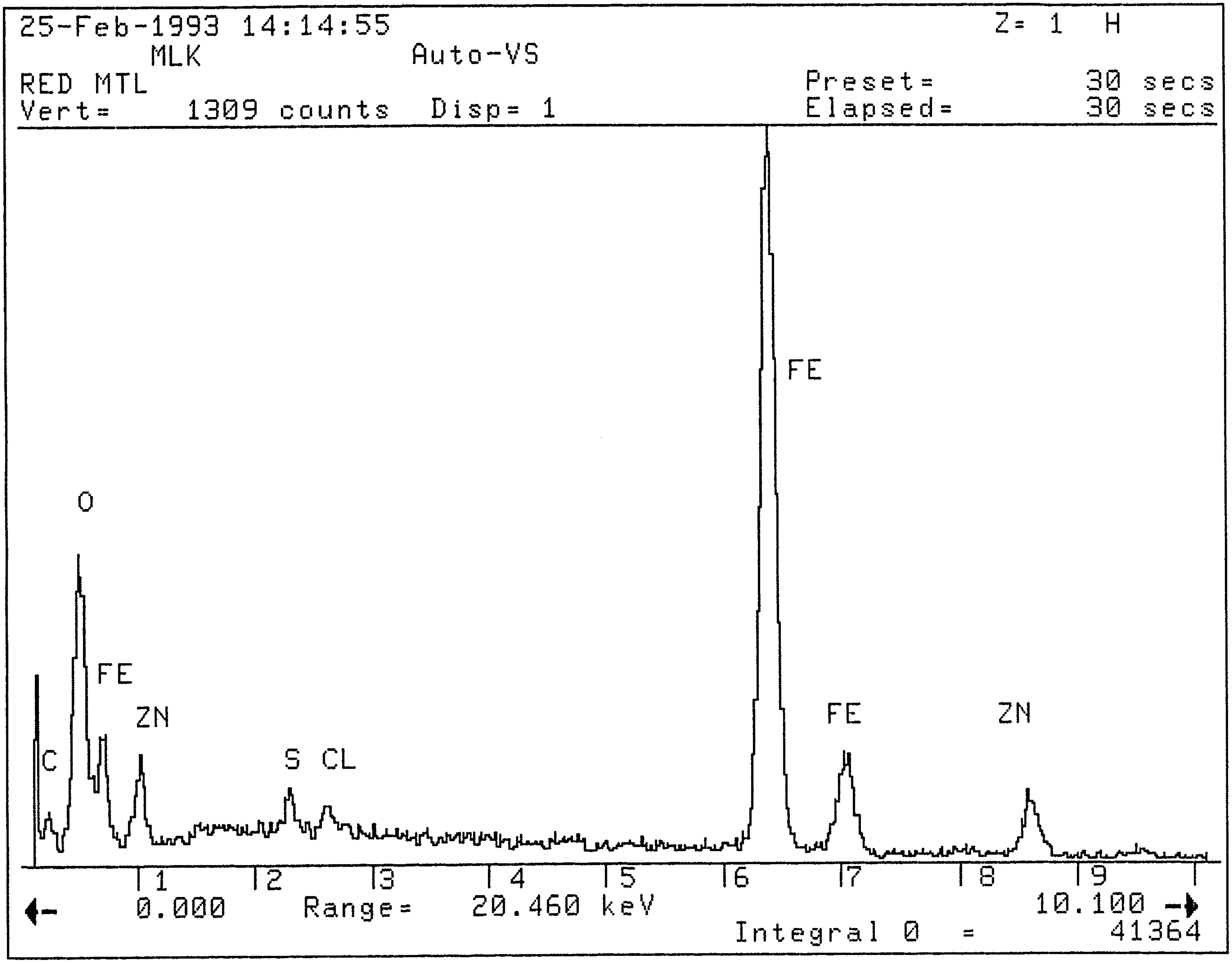

Fig. 83 EDS spectrum of rust particles

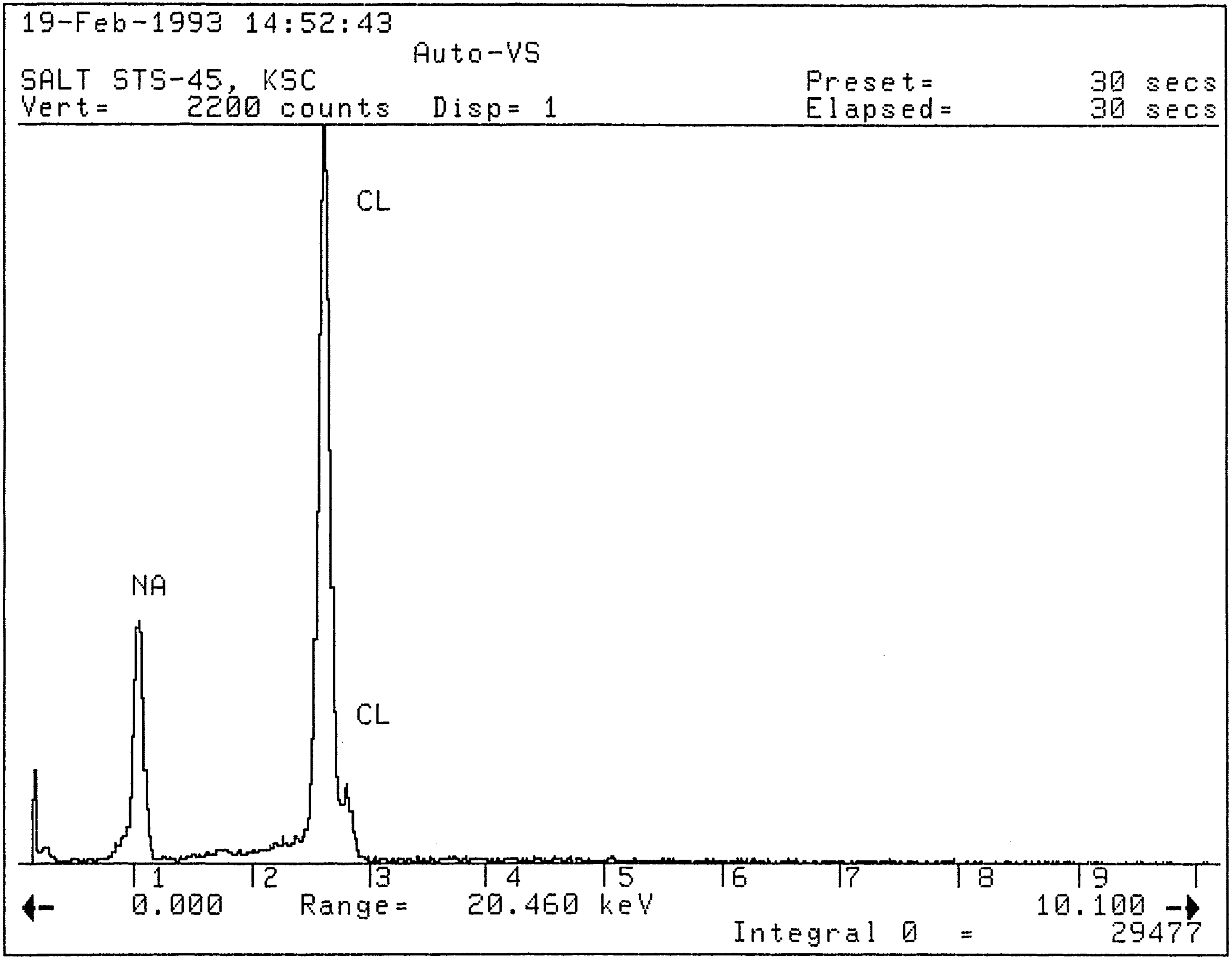

Fig. 84 EDS spectrum of salt

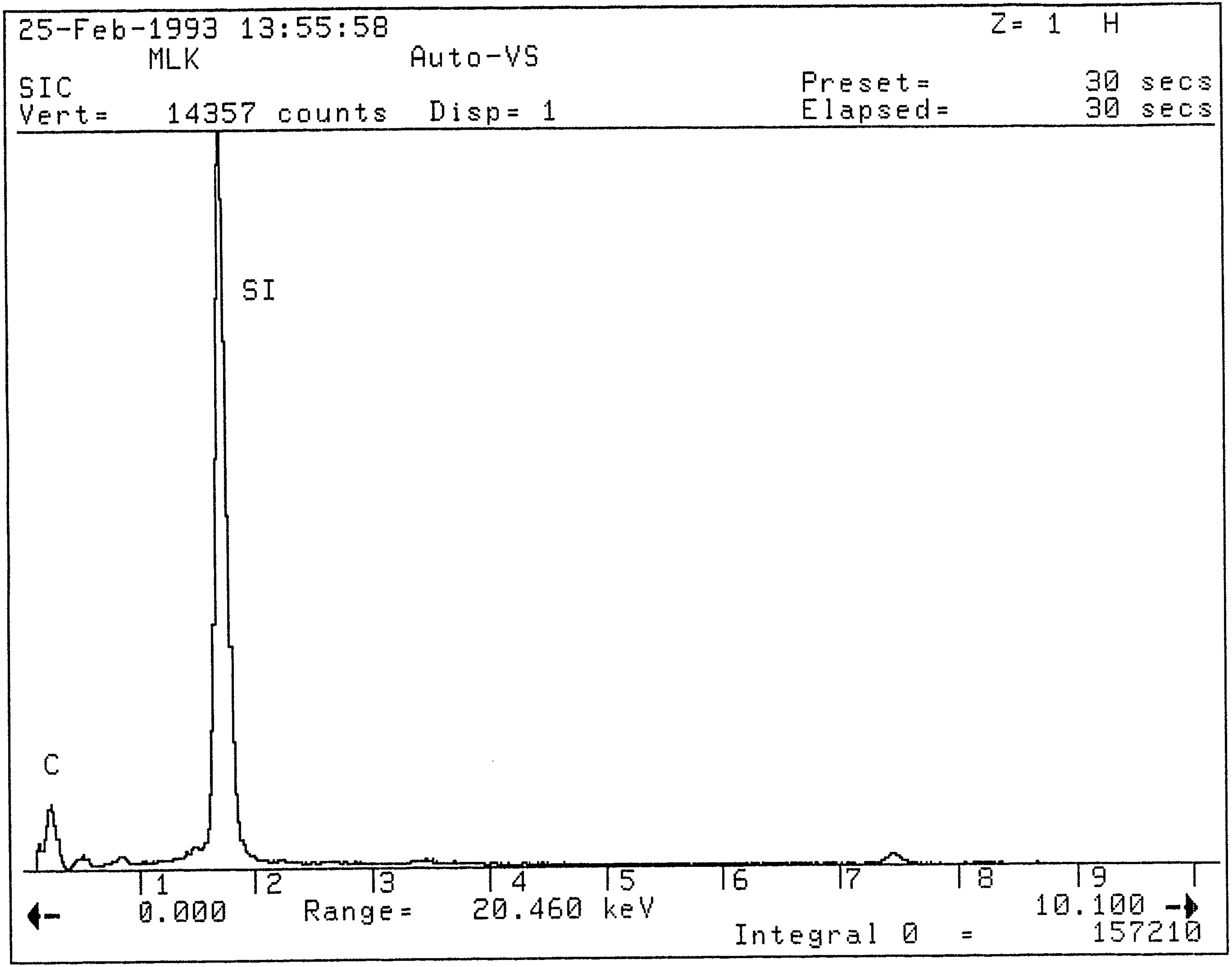

Fig. 85 EDS spectrum of SiC

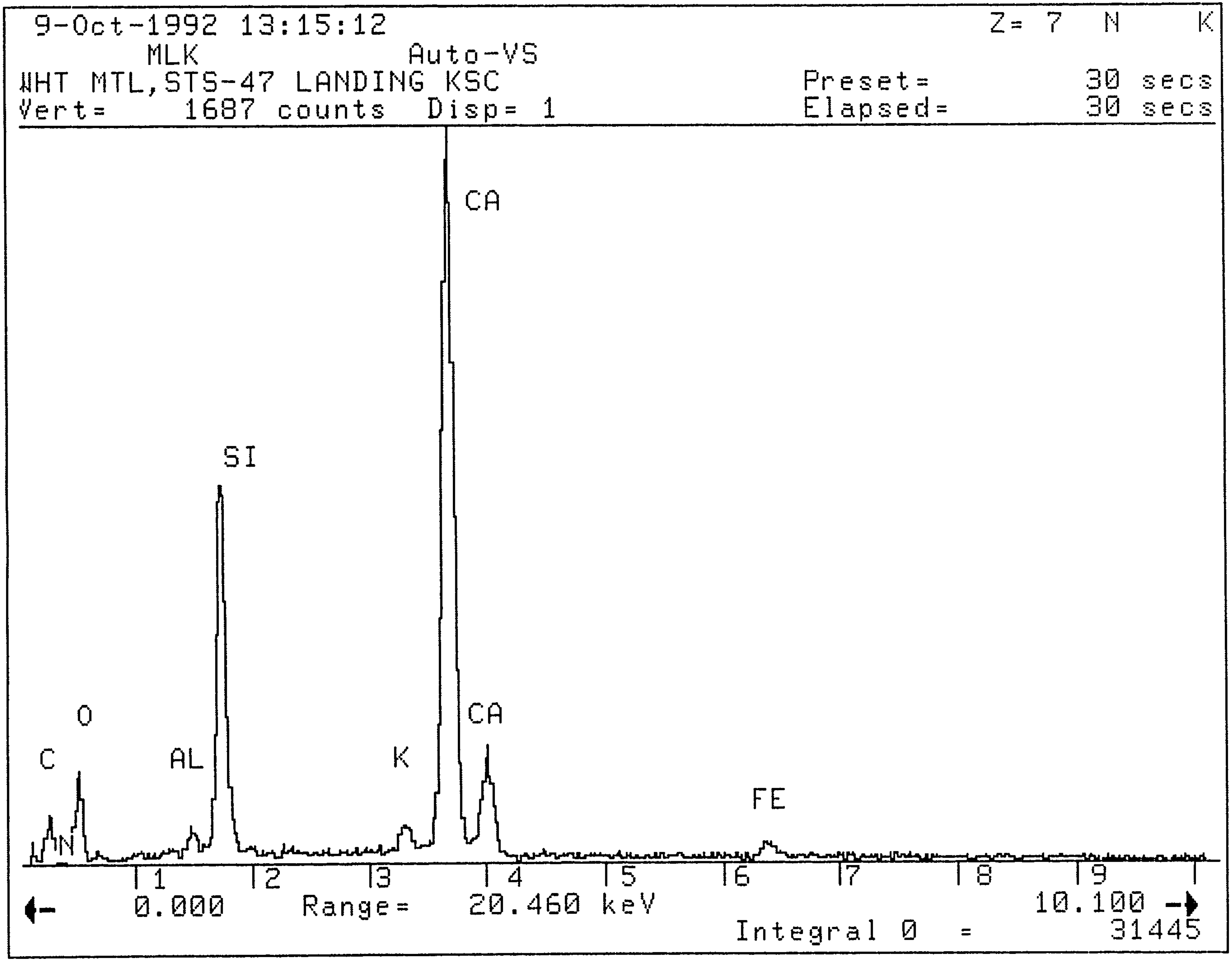

Fig. 86 EDS spectrum of white material

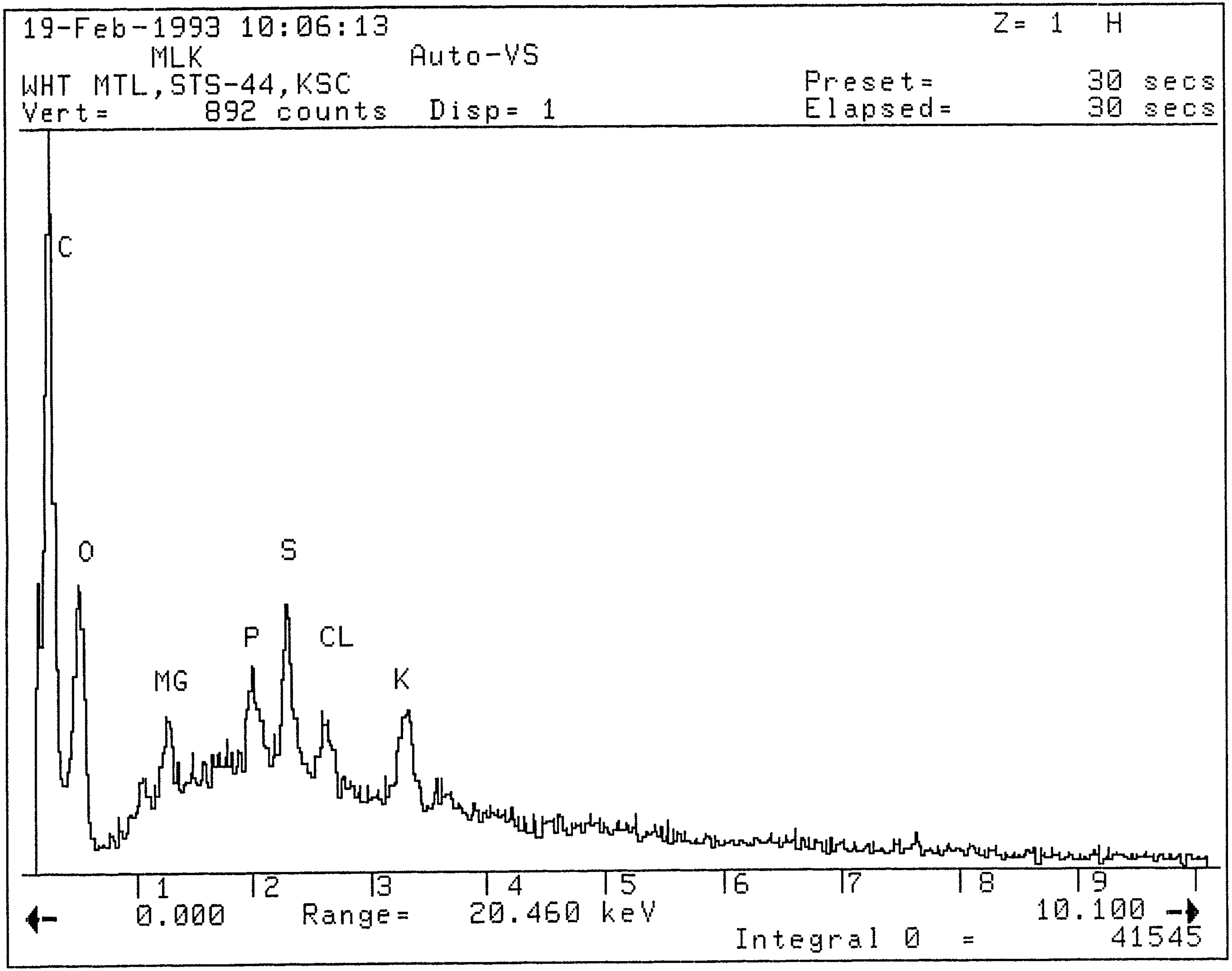

Fig. 87 EDS spectrum of white material

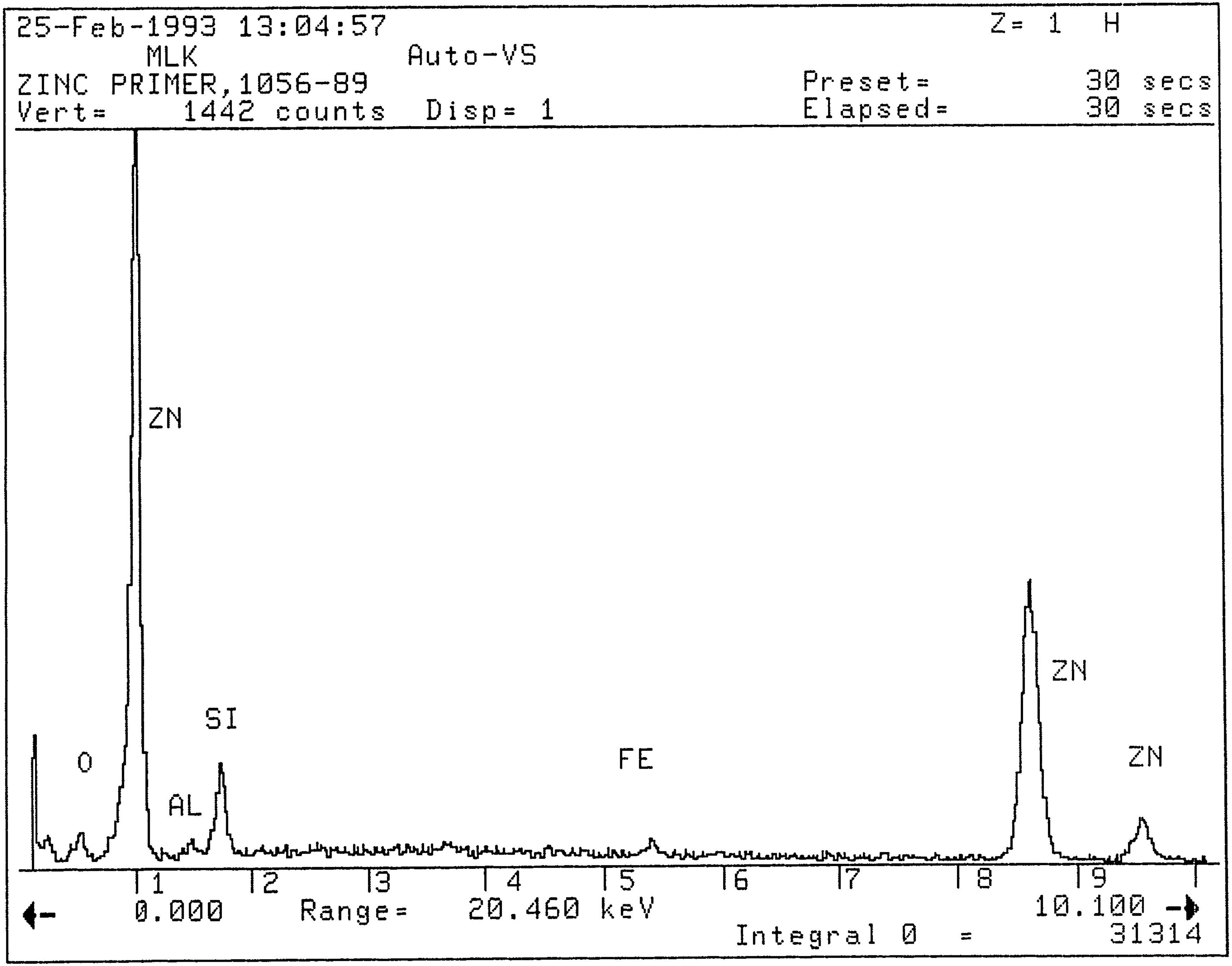

Fig. 88 EDS spectrum of zinc primer

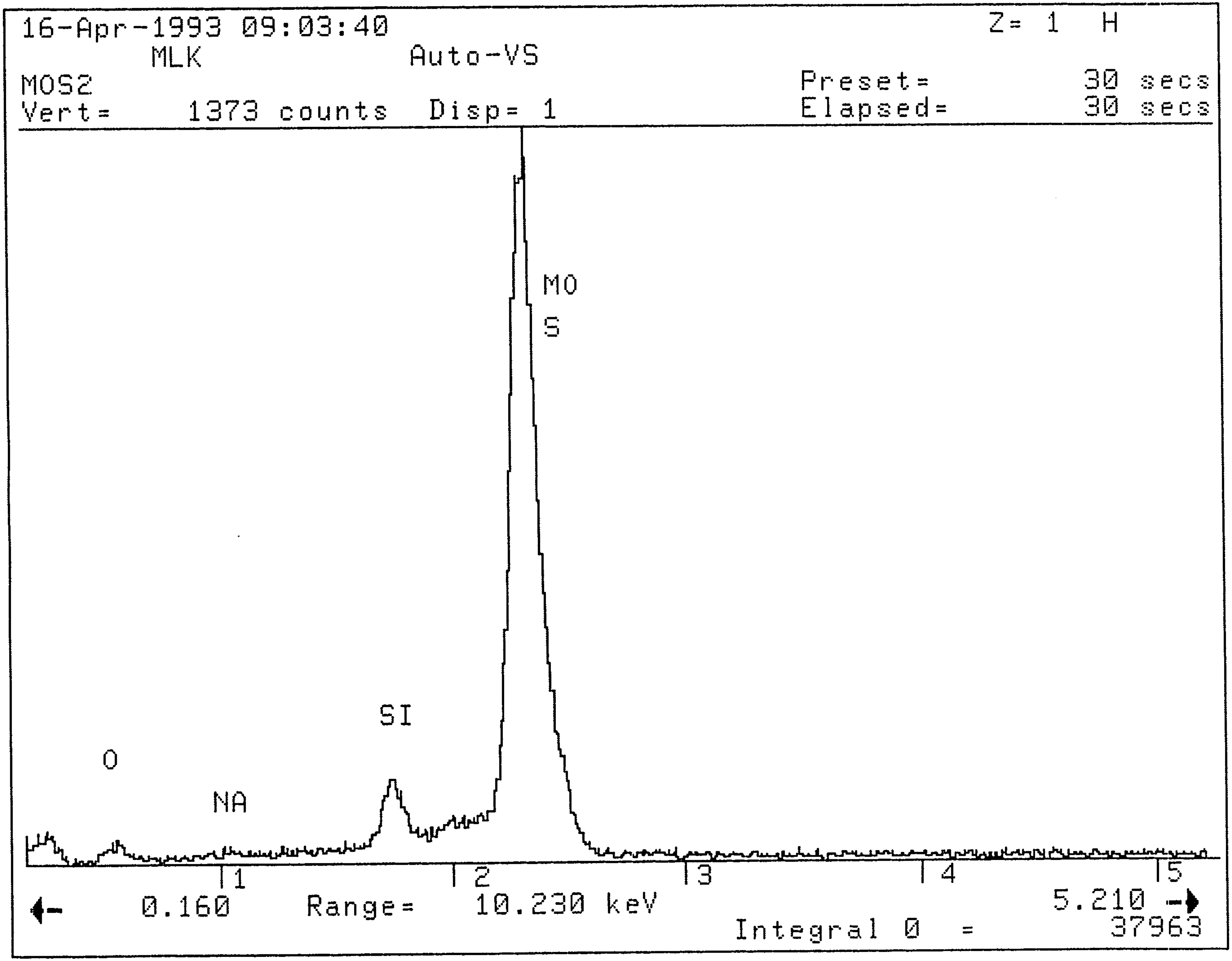

Fig. 89 EDS spectrum of MoS_2

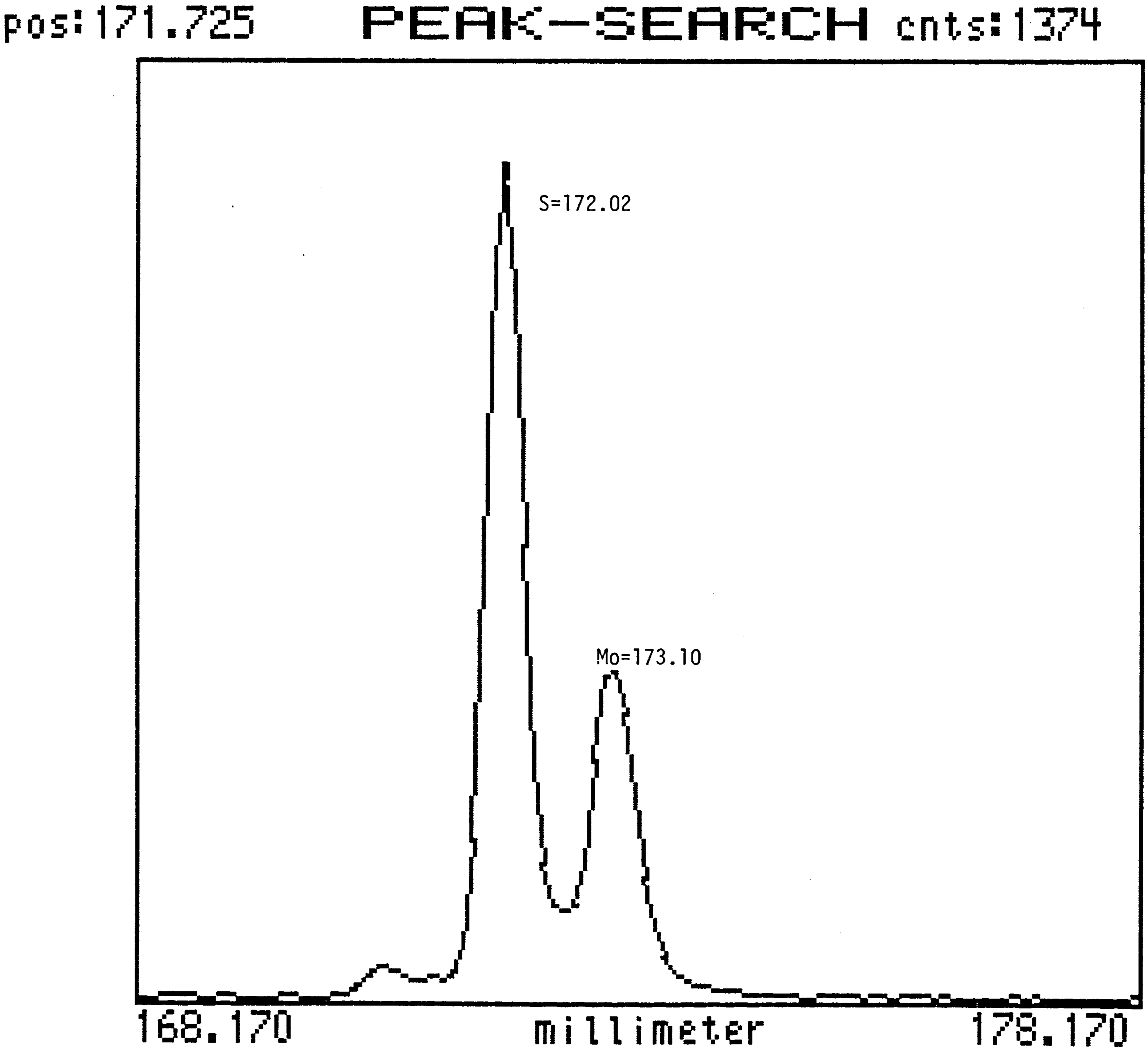

Fig. 90 Wavelength Dispersive Spectrometry (WDS) spectrum of MoS_2

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12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

Debris samples collected from various Space Shuttle systems have been submitted to the Microchemical Analysis Branch. This investigation was initiated to develop optimal techniques for the analysis of debris. Optical microscopy provides information about the morphology and size of crystallites, particle sizes, amorphous phases, glass phases and poorly crystallized materials. Scanning electron microscopy with energy dispersive spectrometry is utilized for information on surface morphology and qualitative elemental content of debris. Analytical electron microscopy with wavelength dispersive spectrometry provides information on the quantitative elemental content of debris.

14. SUBJECT TERMS
Debris, Space Shuttle, Scanning Electron Microscope, Energy Dispersive Spectrometer

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