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SCANNING X-RAY APPARATUS FOR TEXTURE MAPPING BY ENERGY DISPERSIVE DIFFRACTION

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A scanning x-ray apparatus with energy dispersive (= ED) diffraction has been developed [1] which allows point-by-point imaging of a sample surface in the "light" of a selected {hkl} = h reflection (texture mapping), or of an x-ray fluorescence line which is characteristic for the elemental composition (x-ray fluorescence mapping). Texture and XRF mapping are performed in a similar way as conventional mapping of element distributions in SEM. The sample, however, is scanned by a mechanical movement of the stage under the stationary beam spot. The main system components are (Fig. 1):

1) unfiltered ("white") x-ray source from a small-focus x-ray tube,
2) collimator diaphragms for the formation of a small primary beam spot,
3) computer controlled x-y sample stage for unattended scanning,
4) solid state Si(Li) detector with collimator.

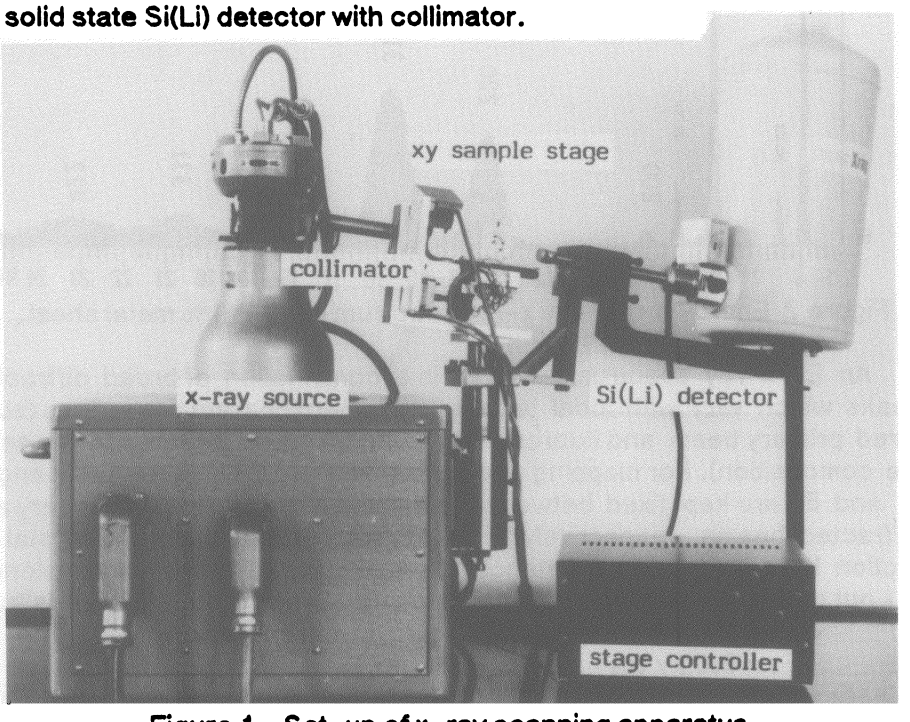

Figure 1 Set-up of x-ray scanning apparatus.

The spectrometer is operated in an air path. This is adequate for all elements above Ar, but vacuum or a helium purge will be required for the detection of light elements or diffraction with photon energies below about 3 keV (corresponding to $\lambda > 0.4$ nm). The minicomputer of the ED system (TRACOR TN 2000) is used to control the x-y raster scan, the acquisition and evaluation of spectral data, and the display of texture maps.

Lateral resolution (0.2 mm with the present system) is limited by the size of the primary beam spot in the sample surface. Since no conventional lenses are available for white x-ray radiation, the spot size is adjusted by selection of collimator diaphragms, in conformity with the grain size and travel steps of the sample stage. Spots less than 0.2 mm in diameter are not yet practicable, due to the low source brightness. An angular divergence of about ± 2° is adequate for most texture measurements, whereas XRF mapping is not affected by the beam aperture. An increase in source brightness is expected from a glass capillary collimator [2), 3), 4)] which would enable a higher resolution or shorter acquisition time.

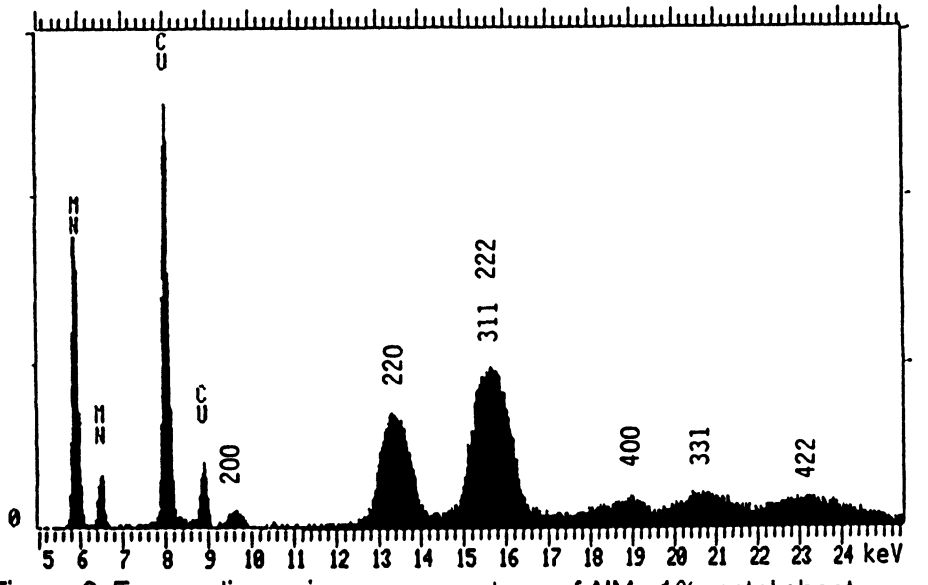

Figure 2 Energy dispersive x-ray spectrum of AlMn 1% metal sheet.

An ED x-ray spectrum (Fig. 2) is a combination of broad diffraction peaks which vary with local texture, and sharp characteristic lines (scattered primary beam and fluorescence intensity being affected by the sample composition). For mapping a texture field with ED diffraction, the angles δ_1 and δ_2 are kept fixed between the sample surface and the primary and diffracted beams, respectively (Fig.3). Grains of an appropriate crystal direction $\mathfrak{h}$ and interplanar distance, $d_{\mathfrak{h}}$, diffract partial rays of wavelength $\lambda_{\mathfrak{h}}$ out of the "white" primary beam such that Bragg's equation holds true:

(1) $\lambda_{\mathfrak{h}} = 2 * d_{\mathfrak{h}} * \sin \Theta$, or in energy units:

(2) $E_{\mathfrak{h}} = 0.62 / (d_{\mathfrak{h}} * \sin \Theta)$ keV * nm.

$\Theta = (\delta_1 + \delta_2)/2$ stands for the Bragg angle. When changing the angle from Θ to Θ', the centroid of diffraction peak, $\mathfrak{h}$, moves from energy channel $E_{\mathfrak{h}}$ to $E_{\mathfrak{h}}'$, according to equation (2). Since energy resolution of solid state Si(Li) or Ge detectors is better than 150 eV at MnKα, overlaps of diffraction peaks with each other or with characteristic lines can be avoided, at least for high symmetry crystals and low index reflections, by an adequate setting of Θ.

The diffracted beams of various crystal directions, h, all belong to the same points (α, ß) on the h pole figures, since they are all collected at the same angle, Θ. Setting $\delta_1 = \delta_2$ as indicated in Fig. 3, only grains with hkl lattice planes parallel with the sample surface can diffract into the detector and hence only the center points of the pole figures are relevant here. If other pole-figure points than those are of interest, the settings of the angles of incidence and reflection must be adjusted different, and the sample has to be rotated around its surface normal or tilted.

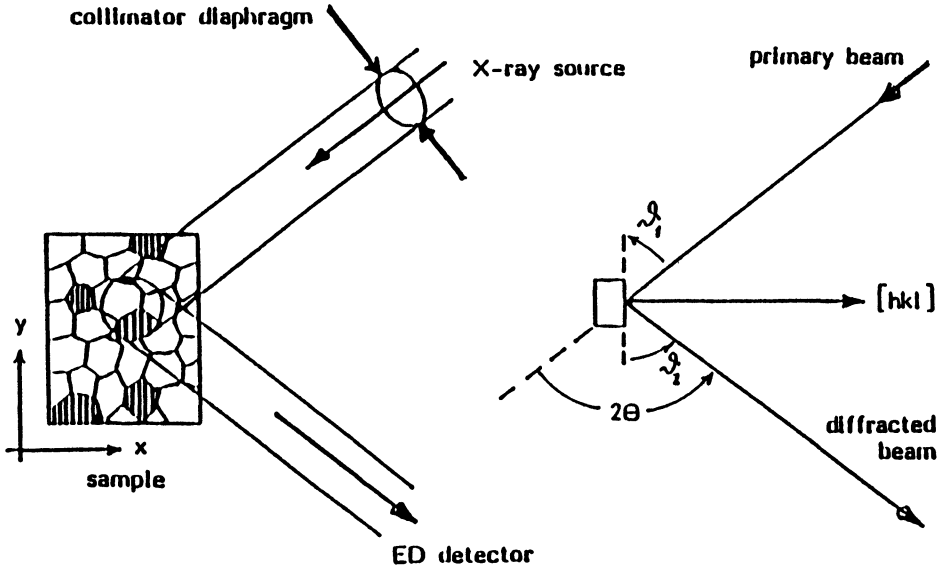

Figure 3 Working principle of x-ray scanning apparatus for texture mapping.

The intensity value detected in a window with centroid E_h is a measure of the volume fraction of grains which have the appropriate orientation and are illuminated by the primary beam spot. This value is taken, after applying some corrections, to paint the corresponding pixel on the monitor screen in a colour out of a scale which is specific for the pole density (Fig. 4).

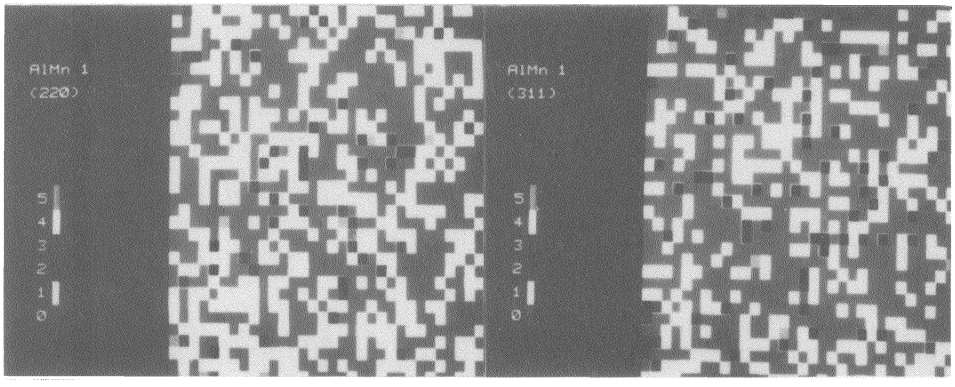

Figure 4 Texture maps of cross-rolled AlMn 1% sheet.

For x-ray fluorescence mapping, a dedicated system has been developed by KEVEX Corp.[5]. Due to the fixed angular geometry, however, this apparatus is not suited for texture mapping.

Conclusion

The scanning method for texture mapping is superior to conventional monochromatic projection arrangements [6],[7], since

* the apparatus is versatile, and capable of texture mapping, energy dispersive diffraction and x-ray microfluorescence analysis. Approximately the same lateral resolutions are obtained.
* the set-up is simple with independent control of lateral and angular resolutions.
* large sample areas, only limited by the travel of the specimen stage, are imaged in focus.
* several maps from complementing reflections can be recorded simultaneously.
* delicate, non-conducting specimens can be studied.
* it is capable of high elemental sensitivity in microfluorescence analysis.

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