# RBS/CHANNELING, SEM AND LAMMA ANALYSES OF SCANDIUM SINGLE CRYSTALS

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*ABSTRACT* — It is shown how RBS/Channeling, SEM and LAMMA techniques can be combined in the surface characterization and analysis of scandium single crystals for channeling experiments. Scandium oxide layers and iron rich particles have been identified and conclusions about the solid solubility of iron in scandium are given.

## 1 — INTRODUCTION

Lattice location measurements in scandium single crystals are relevant to confirm recent theoretical predictions related with volume effects upon alloying of two transition metals [1], and to explain a very high field gradient measured in a scandium matrix, using an iridium probe produced by osmium implantation [2, 3].

The success of the lattice location studies is, however, very dependent not only on the possibility of growing crystals of high purity and good quality but also on the ability to prepare conveniently the crystal surface so that dechanneling will be minimized.

In order to start a systematic investigation in this metal, two random oriented single crystals acquired from different producers were prepared and studied. Results on the surface characterization of these crystals are given. It is the aim of the present work to show how well known techniques like RBS/channeling and SEM can be complemented with LAMMA analysis to characterize phenomena which appear at the near surface region of the scandium metal during annealing, and after bombardment with the analysing helium beam.

## 2 — EXPERIMENTAL DETAILS

The scandium samples, 1 mm thick disks, were spark cut from two random oriented 6 mm diameter and 1 mm thick scandium single crystals. The sample surface was mechanically polished with diamond paste up to 1/4 $\mu$m. One of the samples was implanted with osmium ions at 80 keV and with a dose of about $10^{14}$ atoms/cm$^2$ at room temperature and at a pressure of $5 \times 10^{-6}$ mbar. Annealing treatments were carried out in an oven at a pressure of about $2 \times 10^{-6}$ mbar. Different temperatures and times for the annealing have been tested. The best conditions were found to be an annealing temperature of 800°C during three hours.

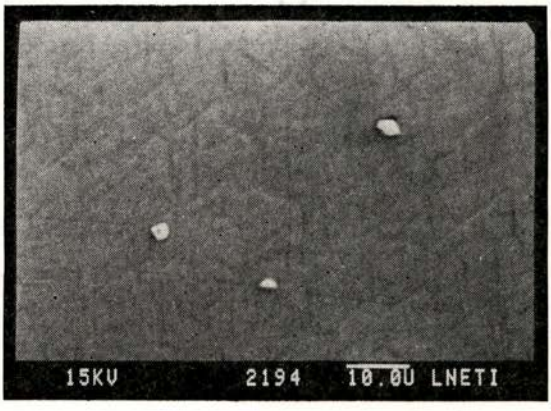
In one case a short time annealing of about 10 min has been tried at 1280°C. Since at this temperature the scandium vapour pressure is much higher than $10^{-6}$ mbar, the scandium contact surface reacted with the alumina crucible and formed a melted layer which has been analysed with the SEM technique. In order to analyse the bulk material this layer has been removed and, after polishment, a layered structure has been observed and completely characterized by RBS, SEM and LAMMA analyses. Details on these techniques are described elsewhere [4, 5].

## 3 — RESULTS

Although the samples of both crystals showed quite different purity (Fig. 1), reasonable channeling quality could only be obtained

after annealing at 800°C for three hours as presented in Fig. 2 and Fig. 3. The amount of particles at the surface of the annealed crystal increased during annealing (Fig. 4). Combining SEM and


a)




b)

Fig. 1 — SEM micrographs of two Sc single crystals which were mechanically polished: a) Sc crystal with many iron particles; b) Sc crystal with higher purity.

RBS analyses it has been possible to prove that these particles are enriched in iron. The RBS spectra confirm also the increase of iron at the surface after annealing treatment. The SEM analysis of the helium irradiated region is presented in Fig. 5. Bubbles have been formed during annealing and large iron rich crystals were observed not far from the bubbles. In one case, evidence could be found for the formation of an aglomerate of small iron rich

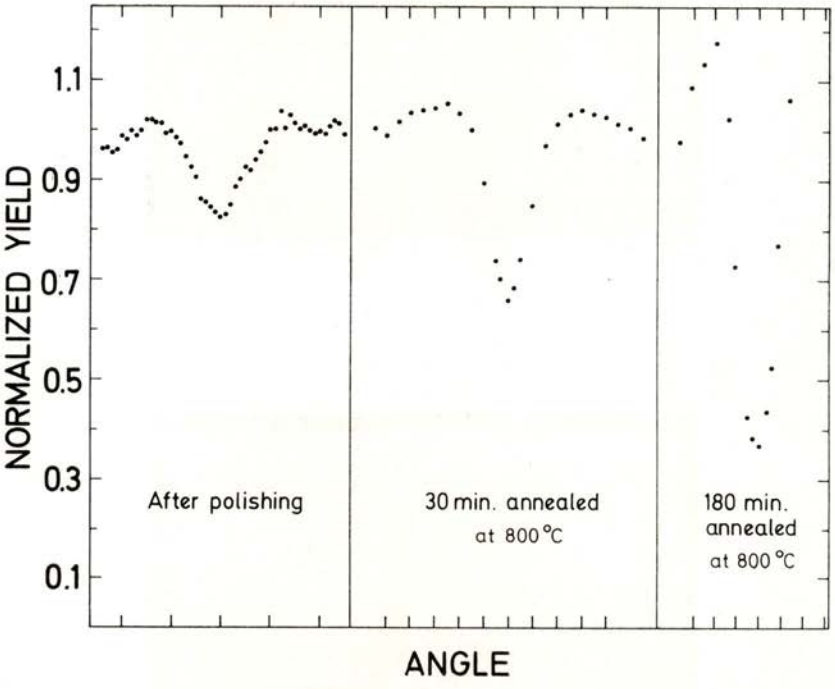



Fig. 2 — (0001) planar scans for a Sc single crystal.

particles (Fig. 6). In the case shown in Fig. 5 the crystal formed presents well defined surfaces. The composition of these particles can be derived from the SEM spectra of Fig. 7. These particles are, most probably, $FeSc_3$ which is stable up to 800°C according to the phase diagram [6].

The melted surface obtained after annealing at 1280°C and

the corresponding bulk structure observed after the mechanical polishing are shown in Figs. 8 a) and b). RBS analysis confirms that scandium remained iron free. The layered structure could be identified through the LAMMA results presented in Fig. 9 as being alternated scandium and scandium oxide layers. Finally, Fig. 10 and Fig. 11 compare the near surface region of an osmium

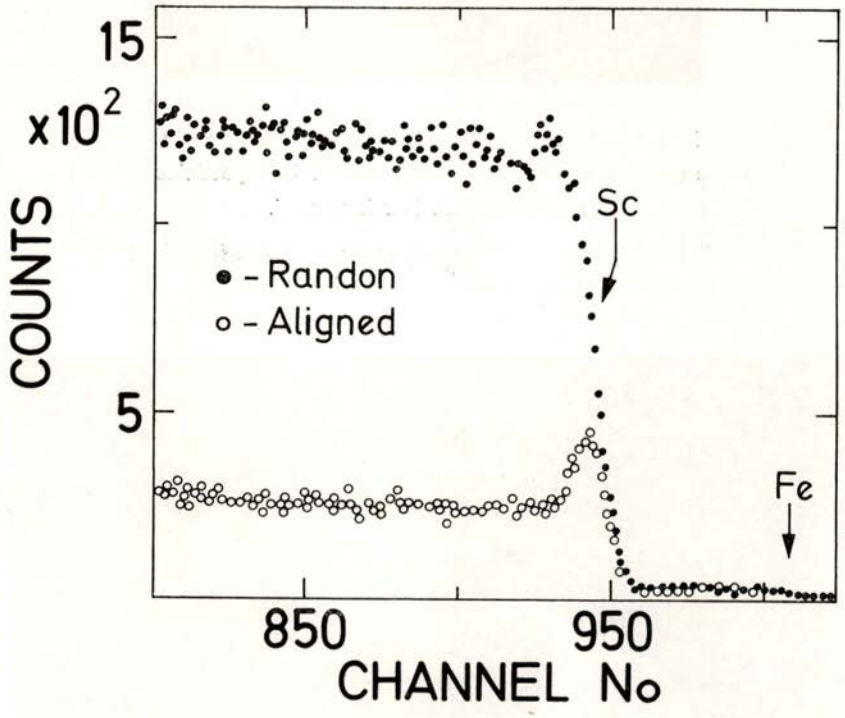



Fig. 3 — Random and aligned $< 11\bar{2}0 >$ RBS spectra after annealing at 800°C for three hours.

implanted and not implanted crystal after annealing. It is clear that dechanneling at the surface increases with the presence of the osmium implanted ions. This can be explained by an enhancement of the surface oxidation during annealing.

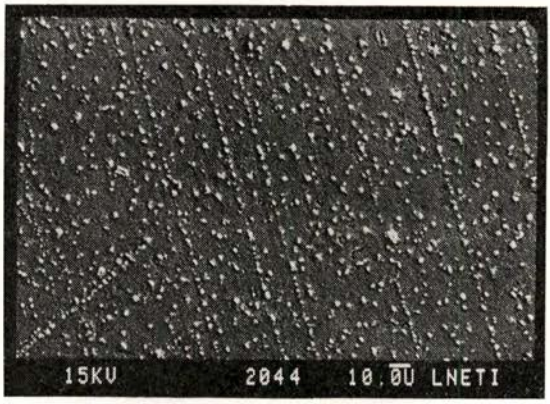



Fig. 4 — SEM micrograph of a Sc single crystal annealed at 800°C. The number of iron particles at the surface has increased.

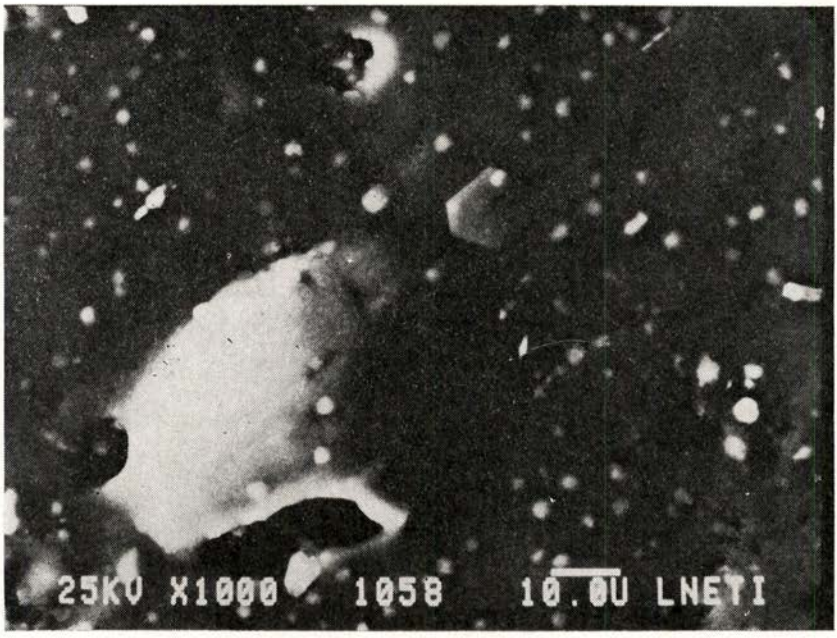



Fig. 5 — SEM micrograph of the helium irradiated region, after annealing.

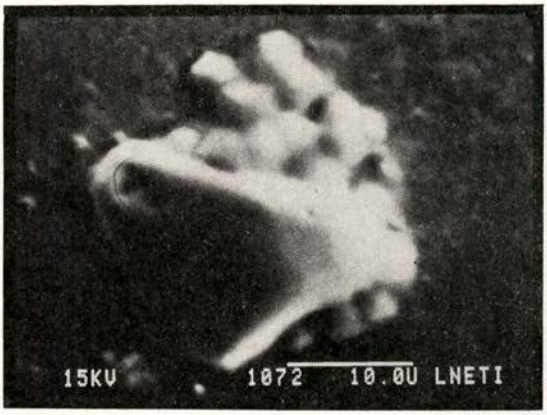



Fig. 6 — SEM micrograph showing an aggregate of iron particles.

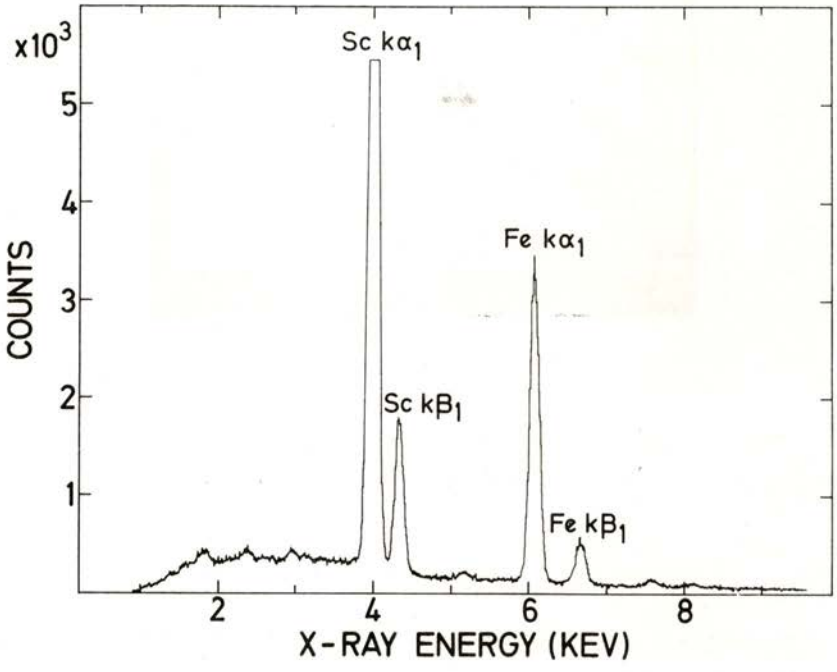
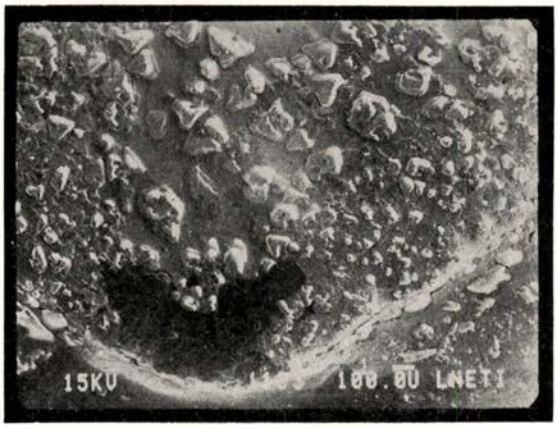
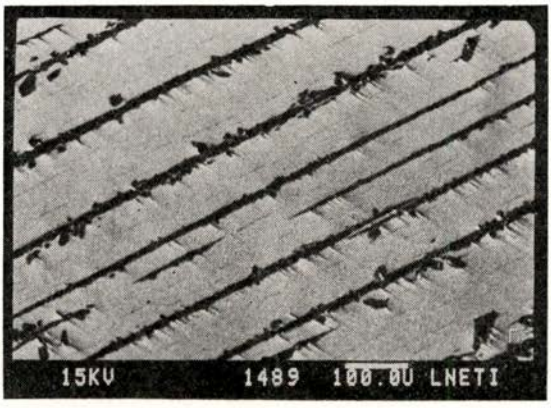



Fig. 7 — X-ray spectrum from the SEM analysis of the iron rich particles.




a)




b)

Fig. 8 — SEM micrograph of the scandium annealed at 1280°C: a) melted surface; b) after polishment.

## 4 — DISCUSSION AND CONCLUSIONS

From the results presented above some significant information on the solid solubility of iron in scandium can be obtained. Solubility of about 1 % to 2 % is predicted as probable in the phase

diagram [6]. The results of the present work do not confirm this prediction and on the contrary they support the idea that the solubility must be very low. This conclusion agrees with the hyperfine interaction measurements where the samples were prepared by melting a small amount of iron with scandium [2, 3]. The very high field gradients measured indicate also that inter-

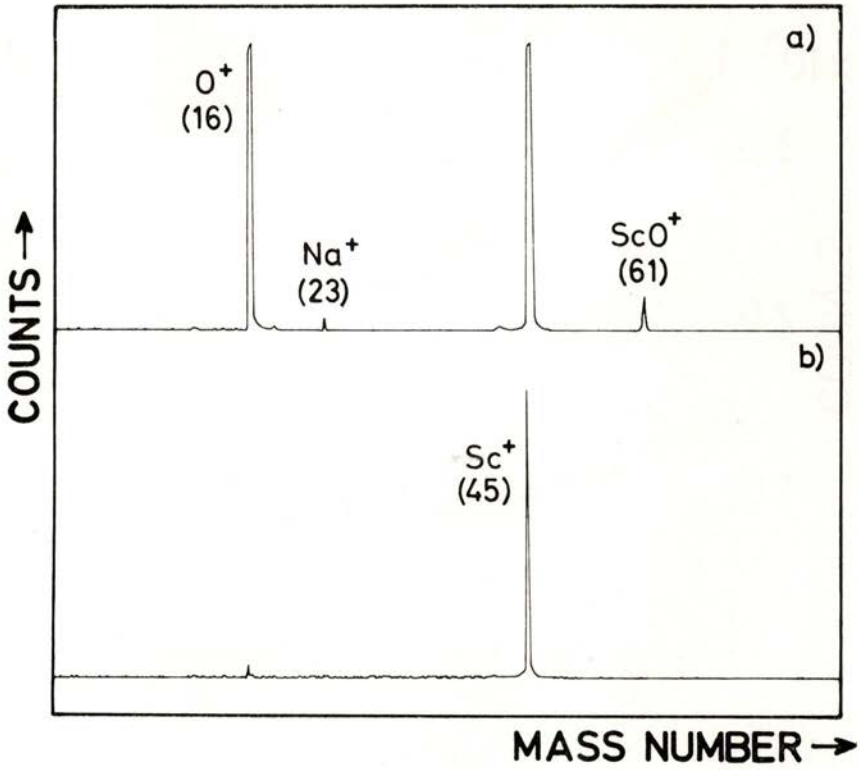



Fig. 9 — LAMMA analysis of the layered structure shown in Fig. 8 b): a) dark lines; b) white zones.

metallic compounds were formed. Our observation that the number of iron rich particles increases with the annealing temperature agrees also with the strong dependence of the hyperfine results on the way the samples are prepared.

The improvement in the channeling quality of the single crystal shown in Fig. 2 might be related with the segregation of the iron content. When big aggregates are formed the scandium lattice shows a higher channeling quality because the number of dislocations in the lattice has decreased. In order to improve the

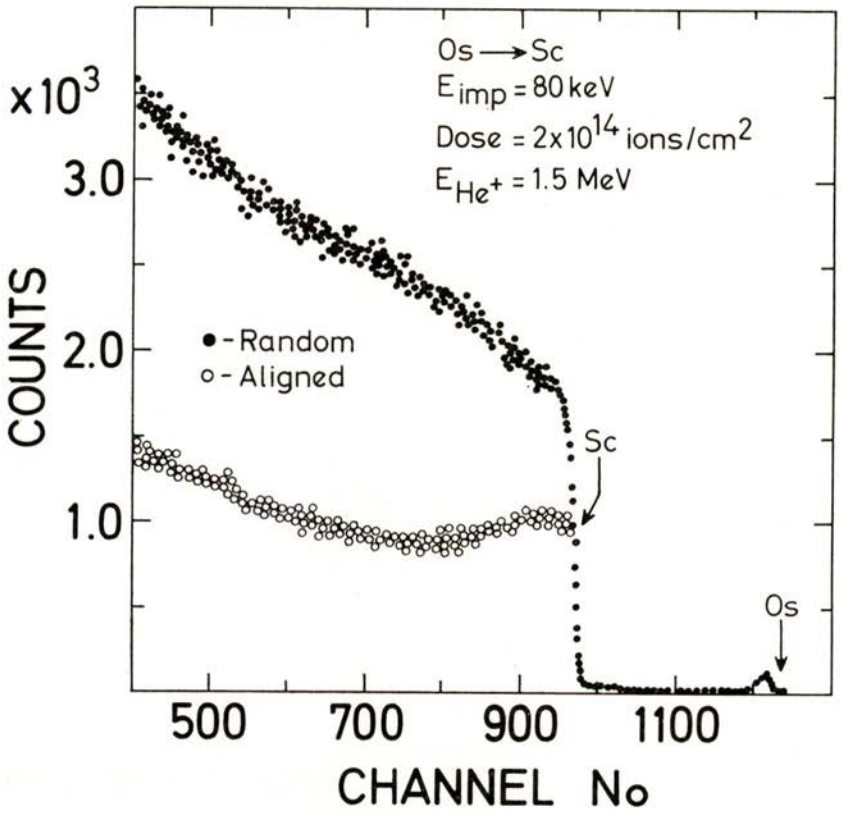



Fig. 10 — Random and aligned RBS spectra of an osmium implanted Sc crystal after annealing.

channeling performance one needs much higher purity and also cleaner surfaces. It could be shown that, at a pressure of $10^{-6}$ mbar, scandium reacts with the residual oxygen and this reaction is enhanced when impurities like osmium are present.

The analysis of the annealed scandium surface in the helium beam spot shows that, probably, helium is not soluble in scandium and, therefore, bubbles are formed when very high doses of helium ions are implanted. Also the radiation induced damage enhances the formation of aggregates of iron rich particles near

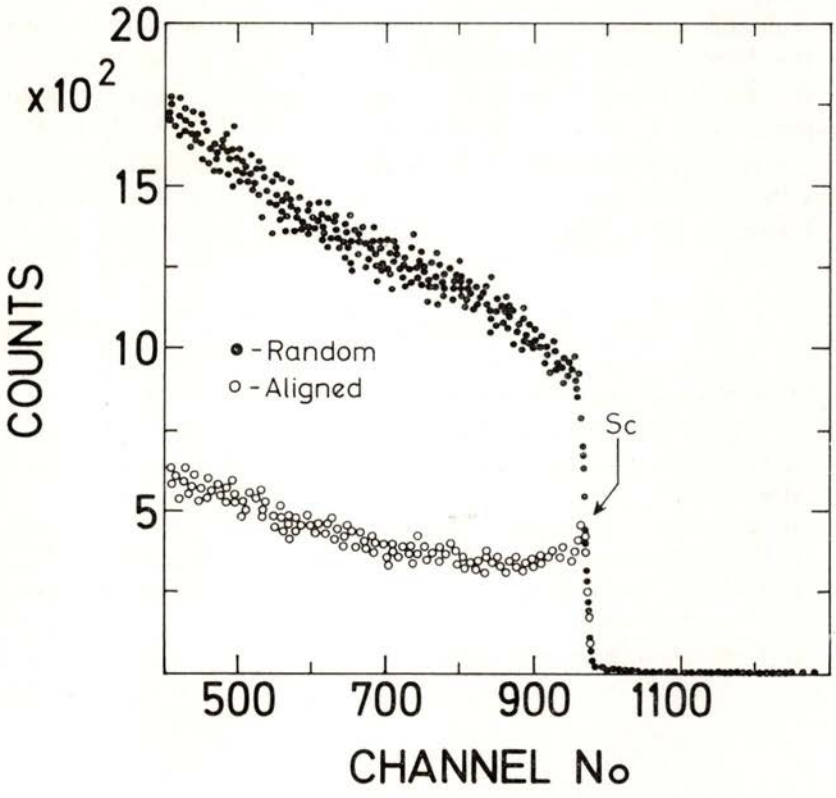



Fig. 11 — Random and aligned RBS spectra of a not implanted Sc crystal after annealing.

the bubbles as is shown in Fig. 6. With these results one can conclude that the RBS analysis using protons might be more useful and that the annealing in a pure helium atmosphere might improve the crystal surface quality.

Short annealings at very high temperature did not improve the channeling quality. They can originate a mixture of scandium-$\alpha$

and scandium-$\beta$, and a contamination of the scandium crystal with scandium oxide layers as those shown and identified with LAMMA. Although the complete segregation of iron has been achieved, the channeling quality of the crystal was hindered by the big amount of $Sc_2O_3$ formed along planar dislocations of the crystal.

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