Fermi National Accelerator Laboratory

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V.V. Baublis, I.V. Ivanov, I.G. Ivochkin, A.V. Khanzadeev, V.V. Kuryatkov, E. Lapin, N.M. Miftakhov, V.F. Morozov, S.R. Novikov, T.J. Prokofieva, V.M. Samsonov and G. Solodov

Petersburg Nuclear Physics Institute
Gatchina, Russia

B.D. Dvornikov, B.B. Loginov, Y.E. Kolesov and A.A. Kochenova

SRI "Electron"
St. Petersburg, Russia

R.A. Carrigan and D. Chen

Fermi National Accelerator Laboratory
P.O. Box 500, Batavia, Illinois 60510

M.N. Strikhanov
Moscow Engineering Physics Institute
Moscow, 115409, Russia

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Fermi National Accelerator Laboratory, Batavia, IL 60510, USA

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ABSTRACT.

A recent experiment has demonstrated spin precession using a bent channeling crystal. In the experiment defect dechanneling was studied and dechanneling from dislocation slip planes was observed for the first time. Information was gained on preparing the crystal detectors. X-ray diffraction techniques were developed for pre-alignment. Ramifications for trans-TeV channeling are explored.

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†   Deceased

1. INTRODUCTION

Recently we studied hyperon spin precession with channeling in bent crystals[1] (Fermilab E-761). Channeling precession may be able to be used to measure the magnetic moments of positively-charged, short-lived particles like charm baryons. For a 375 GeV/c Σ^+ hyperon and a deflection angle of 1.65 mrad (the E-761 parameters), the spin precession angle should be 1 rad.

The E-761 channeling crystals consisted of 400 μm thick, 25 mm by 45 mm oriented silicon plates. Each plate had eight imbedded detector diodes spaced along the 45 mm length. Each detector was 13 mm wide and 2.5 mm along the beam direction.

During the preparation and execution of the experiment many systematic investigations of technical elements related to channeling were carried out. These are reviewed below in sections on selecting, testing, and characterizing channeling crystals, orienting the material, preparing the crystal detectors, and bending and aligning the crystals. (Additional details on these investigations are available in a Fermilab publication[2].)

2. SELECTING, TESTING, AND CHARACTERIZING THE CHANNELING CRYSTALS

Channeling is sensitive to various defects in crystal structure so crystals for experiments must be investigated carefully[3]. Crystal requirements are more critical with increasing energy. In particular, dislocation dechanneling becomes more significant. For example, twice the radius of the dechanneling cylinder[4] due to a dislocation is 6.5 μm at 375 GeV for Si(110) while at E_p = 20 TeV it is 47 μm.

For this experiment we used n-type phosphorus-doped silicon (produced at Zaporozhie Metallurgical Company, Ukraine) with a resistivity in the range 5-20 KΩ*cm produced by float-zone melting. High resistivity silicon was necessary because the crystal also served as a semiconductor with a 400 μm depletion depth. (Note that the high resistivity requirement was not connected with the requirement for an absence of crystal structure defects.) The 62 mm diameter starting ingot was grown with a <111> axis along the ingot axis. Our measurements showed the ingot resistivity variation radially was less than 35% while along the axis the variation was in the range 5-20 KΩ*cm. According to the manufacturer, the minority carrier lifetime ranged from 2200 to 2400 μs. The manufacturer also specified that the oxygen and carbon concentrations were less than 10^{+16} cm^{-3}.

Wafers 450 μm thick with their faces parallel to a (111) plane were investigated for defects. A chemical selective-

etching technique was used to reveal dislocations and clusters while a new Si-H film decoration method (FDT below) was used to observe dislocations, clusters, impurities, and structure defects. Chemical etching showed that none of the starting wafers had any dislocations appearing at their surfaces.

The new FDT has already proved effective for the analysis of crystals used in photo-electrical device fabrication[5]. The method consists of forming both a macro and micro pattern due to color contrast produced by the reaction between Si and the decorating solution. The reaction rate is sensitive to the structure homogeneity of the silicon surface and has different values in regions that have defects or are defect-free. Variations over regions with different inhomogeneities can be evaluated by the color differences of the reaction products. These arise from the interference of the reflected light from the internal and external boundary of the silicon-film system.

The film substance is more chemically active than the bulk silicon due to Si-H bonds. It is easily removed from the surface without material destruction except for a thin silicon layer of 600-800 angstroms involved in the film formation. The FDT method has an advantage over the widely-used selective etching technique due to its high sensitivity. It is also non-destructive. Wafers examined in such a way can be used later for device production.

The FDT technique confirmed the absence of dislocations in the unprocessed silicon wafers but revealed significant concentrations of point defects and clusters especially for wafers from the higher resistivity (7-20 KΩ*cm) portions of the ingot. For E-761, samples were selected with resistivity in the 5-6 KΩ*cm range.

On the basis of these investigations we believe future experiments on beam extraction from the SSC[6] and LHC[7] accelerators could choose silicon starting material with a resistivity of 1-2 KΩ*cm. It is possible to have silicon of this resistivity without any dislocations and, apparently, with minimal inhomogeneity in the distribution of point defects and clusters. This material is less expensive than silicon in the 10 KΩ*cm range.

It is possible to miss internal defects which have not appeared at the plate surface. During the experiment we discovered significant dechanneling at the downstream end of one crystal. Afterward this plate was analyzed using the Si-H technique. A cluster of macro-defects and inhomogeneities from impurities were found near the downstream end. Defect studies using the two-crystal diffraction spectrometer at PNPI confirmed the crystal structure distortion in that part of the crystal. The two-crystal diffraction line widths at two different points along the crystal are shown in Fig. 1. The diffraction peak in

the downstream region was about four times wider than in the upstream portion of the crystal.

The crystals were also reanalyzed with the film decoration technique. Evidence for many new macro-defects appeared on the edges of the plates. Fig. 2 shows the film grown on the surface of one of the crystals after chemical etching of a 30 μm layer. The photograph reveals structure defects such as slip lines. The slip lines[8] are connected with the existence of edge dislocations and their slip at the (111) planes in the <110> directions. The slip of the dislocations results in the formation of steps on the crystal surface. These steps are exposed on the film as the slip lines spaced 60° apart and aligned with the <110> directions. The orientation of these lines agrees with the crystal orientation.

Many of the slip lines were around the edges of the crystals. The edges of the unprocessed wafers probably served as slip line sources. Mechanical disturbances connected with the preparation of the wafers can serve as sources of slip dislocations under high temperature conditions. Note that crystals free of slip lines placed in a bend jig showed no evidence of slip lines resulting from the bending.

3. ORIENTING THE CRYSTALS

The magnetic moment experiment required that the (111) plane be closely aligned with the major crystal face so the crystal planes were oriented carefully. Accurate crystallographic plane orientation is important for many channeling experiments since there are a variety of edge effects or ways particles can be lost out of the sides. For example, if θ, the angle between the major crystal surface and the crystal plane for bending, is $3*10^{-4}$ rad, planes leak out of the plate sides for a 27 μm band in the 45 mm long crystal resulting in a 7% non-functional region for the 400 μm thick crystal. As a result of this non-functioning region, feeding-in (volume capture) and loss of particles from the channels will occur along almost the entire length of the crystal, imitating real feeding-in due to multiple scattering and dechanneling.

An X-ray spectrometer was used to determine the ingot orientation to an accuracy of $(1-2)*10^{-4}$ rad, limited by the precision of the spectrometer. The plates were polished to a thickness of 400±20 μm with less than ±1 μm variation to maintain plate parallelism in the goniometer multiple plate bending system. After the orientation process the crystallographic alignment parameters for our samples were: $\theta < 5*10^{-4}$ rad, $\eta < 5*10^{-4}$ rad, and $\phi = 7 \pm 0.5°$, where η is the tilt angle of the crystal major face relative to the bending plane in the upstream face and ϕ is the angle between the (110) plane perpendicular to the bending (111) plane and the side of the crystal.

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While this orientation accuracy for Θ was satisfactory at 375 GeV, it would not work for LHC beam extraction. The accuracy can be improved by using a precise two-crystal diffraction device in which the first crystal serves as a monochromator and the second is the crystal under investigation. With an optical auto-collimator it would be possible to check the position of the crystal surface. Accuracies of less than ±5 μrad could be achieved with this approach.

4. PREPARING THE CRYSTAL DETECTORS

Semiconductor detectors were prepared in the crystals after they were oriented. Ten sample wafers were selected based on the reverse currents of the diodes. The detectors were prepared at the research institute and production facility "Electron" (St. Petersburg, Russia) with the technique that is normally employed for production of electronic components from low resistivity silicon technology. P-n junctions were produced by boron diffusion from a boron-doped, poly-silicon layer $Si^*(B)$. The n^+-layer was made by phosphorus implantation on the obverse side with an implantation energy of 110 KeV and a fluence of $3.0*10^{15}$ particles/cm^2. An aluminum layer with a thickness of about 1 μm was evaporated in vacuum on both sides for contacts.

The electronic response of these detectors was unexpected. The electronic behavior of the silicon was significantly changed in the process of preparing the detectors. While the resistivity of the starting material was in the 5-6 KΩ*cm range, analysis of the current-voltage and capacitance-voltage response curves indicated that the resistivity of the materials after processing was lowered to the 0.5-1.0 KΩ*cm level. This lower resistivity resulted in a smaller depletion depth of 140 to 200 μm for an applied 150 V reverse bias.

The low resistivity and the resulting incomplete depletion was a problem, decreasing the useful thickness of the plates by a factor 2-3. The signals from particles passing through the undepleted layer had anomalously small amplitudes. Since the E-761 apparatus had a precise microstrip tracking system, it was possible to reject events from the undepleted regions of the crystals.

After the experiment was completed an analysis was carried out to determine the cause of the resistivity decrease. New sets of detectors were prepared in new silicon wafers from the same ingot. The material resistivity was monitored after each production stage using Deep Level Transient Spectroscopy (DLTS)[9]. With DLTS it is possible to determine the main parameters of deep energy levels of electrically-active centers at concentrations of ≥ $2*10^{-13}$ atomic percent. The measurements were made using an automatic relaxation DLTS spectrometer developed at the Petersburg Nuclear Physics Institute (PNPI)[10].

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With this spectrometer it is possible to study the relaxation of diode capacity produced by deep levels with time constants from 10^{-4} to 10^{-1} s over a temperature range of 77 to 300 K°.

The preparation involved six technical stages; I) the unprocessed materials, II) oxidation at 1000°C in a "wet" oxygen atmosphere with chlorine dopants for three hours to form a 0.6 μm thick oxide layer for the contact plates, III) a repeat at 1150°C in a dry oxygen atmosphere for one hour to form a 0.2 μm oxide layer to separate the diodes, IV) boron diffusion at 1150°C in an oxygen-nitrogen mixture, V) annealing in a hydrogen atmosphere at 900°C for five minutes, and VI) annealing in H_2 at 450°C for thirty minutes. A significant decrease of resistivity appeared after the V stage due to the appearance of new electrically-active centers with a concentration of approximately 10^{13} cm^{-3}.

The resistivity measurements after the high temperature treatments in stages II-VI of the fabrication are shown in Fig. 3. After stage V a new deep energy level in the DLTS spectrum appeared in all the wafers with a concentration several times more than the dopant concentration. The appearance of this new level resulted in the abrupt decrease of the material resistivity. The concentration of this level is exactly equal to the increase of the electron concentration resulting from doping the samples after the V stage. It is known that atomic hydrogen cannot produce any kinds of deep levels in silicon. However, estimates show that none of the known impurities other than hydrogen can penetrate silicon to depths of 150 - 200 μm in 5 minutes at 900°C. This suggests that the level is a complex between hydrogen and an electrically inactive impurity contained in the unprocessed material.

After this analysis the detector preparation technology was altered. The hydrogen anneal at 900°C was changed to a low temperature anneal in a nitrogen atmosphere. This prevented the creation of the centers and significantly improved the volt-ampere characteristics of the detectors.

5. **THE BENDING AND ALIGNMENT SYSTEM.**

The E-761 bending and alignment device is shown in Fig. 4. This device has several parts including a six-point bend structure which holds the seven crystals, two remotely-controlled mechanisms to change the crystal bending angle for the upstream and downstream ends of the crystals, and a remotely-controlled goniometer that rotates the bending device around a horizontal axis.

The upper and lower crystals were respectively bent down and up to observe spin precession in opposite directions. The center crystal had a fixed support and remained flat. The requirement for separate bending devices for the upstream and downstream

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segments was caused by the experiment geometry. It was necessary to orient the crystal front ends so they pointed at the target while the deflected beam also had to pass through a hole in a calorimeter.

A two-crystal x-ray diffraction spectrometer geometry was used to orient the crystals before the experiment. This spectrometer also measured the curvature of the bent crystals. The entire goniometer bending array was placed at the normal location of the first crystal in a two-crystal spectrometer. Bragg diffraction off the (111) planes was used. The (111) plane was used as the Laue-reflection plane for the crystal-analyzer so that the two-crystal spectrometer had no dispersion and the width and shape of the diffraction line depended only on the conditions for diffraction.

Studies were also carried out on the distortion of the bent crystals using the ANSYS finite element program and direct observation of channeled particle directions with the spectrometers in the experiment. These studies showed the distortions (illustrated in Fig. 5) were small. Most of the change of curvature came from the middle sections of the crystals. The bending angle variation ranged from 1.5 mrad in the middle to 1.618 mrad at the crystal edge.

The crystals exhibited a behavior like that expected for a twisted crystal so that the channeling angle changed along the front edge of a crystal. It is possible a screw-like twist could have been introduced in the crystal by the shimming that was used. This would result in the deflection angle (DTY below) changing with x.

These distortions were measured using the fine resolution of the E-761 spectrometer. ANSYS shows the maximum variation of the entrance angle (TY1) vs x over a 4 mm active region in x is about 4 μrad. The actual variation over the interval was 40 μrad so most of the variation must have been due to twist-like effects. The bend (DTY) distribution should exhibit more curvature. The observed variation over the 0.4 cm x region used for the experiment is consistent with the ANSYS simulation.

6.   **SUMMARY**

These studies for the spin precession experiment have led to progress in several areas. For the first time sophisticated x-ray pre-alignment schemes were used to set crystal bending angles and orientations. New tools were developed for studies of defects in channeling crystals. Dechanneling from dislocation slip planes was observed for the first time. New insights were gained concerning techniques for preparing detectors in channeling crystals. System spatial and angular resolution was good enough to observe crystal distortions and see their impacts.

The experiment has shown that sophisticated technical and crystal quality issues can be solved for channeling applications in the TeV range. It has also pointed the way to investigating these issues at levels required for channeling applications in the 10 TeV regime prior to the availability of beams.

FIGURES:

1. Two-crystal spectrometer lines for two positions of the x-ray beam on the crystal surface (L=0 is upstream).

2. Photograph of the Si-H films grown on the surface of a crystal after chemical etching of a 30 μm thick layer. The films reveal such structure defects as foliation and slip lines.

3. Variation of resistivity and the associated electron concentration deduced from the resistivity for two wafers (•, ▲) with original resistivities in the range of 2 and 4 KΩ*cm, after each of the treatment stages I - VI (see text).

4. The goniometer has several principal parts: (1) seven silicon crystals, (2) crystal support structure, (3) mechanism for manual rotation relative to other parts of the bending device, (4) remotely-controlled mechanisms to change the crystal bending angle for the downstream and upstream ends of the crystals, (5) pre-amplifier mount for the semiconductor detectors on the surface of the crystal, and (6) a goniometer that rotates the bending device around a horizontal axis.

5. XY cross-sections of the crystal for the upstream end and the mid-section. Y values are in μm. Most of the change of curvature comes from the middle section.

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