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NIST XRD Standard Reference Materials: Their Characterization and Uses

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SRM production and certification

Standard Reference Materials (SRMs) from the National Institute of Standards and Technology (NIST) are designed to increase the accuracy and precision of measurements which are pertinent to science and industry. Powder diffraction SRMs consist of stable materials which have one or more diffraction properties measured and certified. The success of an SRM is dependent on the material and certification measurements being of such quality that the accuracy and precision of the certified values allows for the calibration of subsequent measurements made by the user community. The standardization of such measurements is achieved by the performance of a material rather than the standardization of equipment design and/or measurement procedures.

The certification of a NIST SRM begins with the isolation of a measurement error which may be corrected with the proper use of a standard. A preliminary study is undertaken to determine the feasibility of producing an SRM which will address the problem. In addition, the potential demand for, and impact of, the SRM is evaluated. A proposal for the production of the SRM is then submitted to the Standard Reference Materials Program (SRMP) for review and subsequent funding. The SRMP of NIST operates as a non-profit business with the costs of developing and producing an SRM being fully recovered by the sale of that SRM. Thus the demonstration of the demand for the SRM is critical to a favorable response.

Once the decision to produce the SRM has been reached, an "idealized character" of the SRM material is determined from a review of the literature. The effects of deviations from this "ideal" character exhibited by available materials are the subject of subsequent experiments. The characteristics investigated are those judged as capable of having an effect on the critical property which is at least as large as the smallest conceivable measurement error of that property. The study may include the development of improved measurement techniques which increase the accuracy and precision of the property determinations. Throughout this work the procurement or production of several kilograms of material for the SRM itself is under consideration. The material selected for the SRM must be optimal with respect to the desired property, homogeneous, stable, and of such character as to minimize errors in the measurements made by the end user.

When the required quantity of material has been obtained and verified to be acceptable, it is riffled and bottled by the SRMP. During bottling, a representative sampling of bottles is removed from the stock for certification measurements. The certification process is designed to determine the homogeneity of the material and also determine, via methods traceable to fundamental physical constants, the value of the certified property. However, in the case of certain SRMs, such as the line broadening SRM 660, the desired property does not lend itself to absolute measurement. In such cases the desired property is determined to be appropriate for the demands placed on the SRM by supplemental research or a round robin study, in which case the certification process is used to determine homogeneity only. Modern statistical procedures are used to design and analyze experiments to insure that the measurements are as accurate as possible and the uncertainties fairly reflect the precision of those measurements.

Line position SRMs

The primary line position SRM, silicon SRM 640b, is the most popular of the NIST powder diffraction SRMs. SRM 640b consists of a silicon powder, jet milled to a median particle size of about 5 μm from electronic grade single crystal boules, certified with respect to lattice parameter. A second line position SRM 675, fluorophlogopite, is used for low angle measurements. The popularity of SRM 640b reflects the widespread adoption of the internal and external standard methods for use in lattice parameter measurements. These procedures involve the determination and application of a delta d curve which represents the end result of the various optical aberrations of the particular diffraction equipment. A polynomial is then fitted to the curve and

applied either to subsequent measurements in the case of the external standard, or to the line positions of the unknown with which the SRM has been mixed in the case of the internal standard. The external standard method is less reliable because it cannot account for a possible sample displacement error of the unknown. Adoption of the internal standard method has resulted in consistent and routine measurements of lattice parameter to 1 part in 10^4 (Edmonds *et al.*, 1989).

SRM 660, LaB_6, is certified with respect to lattice parameter. However, its primary virtue is a lack of size- and strain-induced peak broadening. The suitability of this material for the determination of instrumental broadening functions prerequisite for size and strain analysis was determined with a round robin study (Fawcett *et al.*, 1988). The SRM displays evenly spaced, non-overlapping, high intensity diffraction lines whose full-width-half-maximum (FWHM) values measured on fixed slit diffraction equipment closely follow the expected quadratic dependence on 2Θ angle. These properties also render it suitable for use in determining calibration parameters for a Rietveld refinement.

The Rietveld method uses models of the origins of the specific sources of optical aberration to refine the degree to which they contribute to a delta d curve. This technique is applied to data from the unknown only; no standards are used. The delta d curve derived from an unknown may differ significantly from that of an admixed standard due to variations in character between the unknown and the standard. Therefore, an increase in accuracy could be realized with the use of the Rietveld method. The chief factor preventing the realization of this potential is the lack of fully developed models appropriate for conventional divergent beam x-ray equipment. These models will have to address aberrations which are inherently more complex than those associated with parallel beam equipment. In the case of the software Generalized Structure Analysis System (GSAS) (Larson & Von Dreele, 1987), the refined delta d curve is obtained with a specimen transparency and sample shift correction. However, examination of actual delta d curves from equipment of various manufacturers indicates that these corrections alone cannot account for the observed aberration curves.

With the application of the pseudo-Voight profile function of GSAS to conventional x-ray diffraction (XRD) data, it is reasonable to assume that the instrumental contribution is primarily Gaussian, while the sample contribution is Lorentzian. Thus refinements of SRM 660 can be used to obtain a set of initial profile parameters; only the Lorentzian terms are released in subsequent refinements. The value of the Lorentzian terms obtained with SRM 660 are then subtracted from those refined for unknowns in size and strain estimations. In this application the models describing optical aberrations affecting peak position can be tested using a material of known lattice parameter. However, in order to perform this function the lattice parameter of the SRM and the wavelength of the x-rays must be known to an accuracy and precision greater than the error introduced by improper or incomplete model(s) describing the optical parameters.

The wavelength of Cu $K\alpha_1$ radiation has been determined to be 0.15405945 nm by combining single crystal lattice spacing measurements (x-ray/optical interferometry) and diffraction angle measurements (Deslattes & Henins, 1973; Kessler, Deslattes & Henins, 1979). The quoted value is taken from "The 1986 adjustment of the fundamental constants" and is considered accurate to 0.7 part in 10^6 (Cohen & Taylor, 1987). The lattice parameters of the silver and tungsten used as internal standards for the certification of SRMs 660, 640b, and 675 were measured in 1966 with a high precision, temperature controlled, back reflection camera (Swanson, Morris & Evans, 1966). Linkage to the fundamental length standard was through the wavelength of the radiation used.

The procedure by which the lattice parameters of existing SRMs were measured and certified is neither precise nor accurate enough to yield SRMs suitable for development of the Rietveld method as applied to divergent beam x-ray diffraction equipment. A project has begun to develop a new high accuracy/precision lattice parameter SRM(s) linked to the fundamental length standard via procedures which are independent of the x-ray wavelength determination. This project involves the lattice parameter determination of a silicon single crystal relative to the iodine stabilized HeNe laser length standard with an X-Ray Optical Interferometer (XROI). A second experiment will transfer the lattice parameter measurement from the single crystal of silicon to a crystalline powder via x-ray diffraction equipment of parallel beam optics. Supplemental studies will include an investigation of the effects of crystallite size on lattice parameters. It is anticipated that this project will result in an SRM in about 2 years.

Instrument sensitivity SRM

Line position SRMs are used to calibrate equipment with respect to the angular variable of the goniometer. SRM 1976 is a new SRM which is designed to calibrate equipment for the intensity variable as a function of 2Θ angle, i.e., the instrument sensitivity. The need for this SRM was defined by the Jenkins, Schreiner & Dismore (1992) round robin which was, in turn, prompted by the consideration of a new generation of database by the ICDD. The new database will consist of complete

digitized diffraction patterns as opposed to reduced d and I values. The removal of any instrumental aberrations from the reference data reported to the ICDD for use in this database will require as yet undeveloped algorithms. The round robin was used to assess the level of variation in instrument performance, which indicates the degree of difficulty to be faced in the building of the new database. Highly uniform, sintered α alumina, corundum structure, plates were circulated among selected respondents with instructions on the collection of a range of intensity data. Among other things, variations in instrument sensitivity of up to 30% were discovered.

SRM 1976 consists of a sintered α alumina plate originally manufactured as a micro-chip carrier. The plates exhibit considerable, though highly uniform and cylindrically symmetrical, levels of preferred orientation. This cylindrical symmetry allows the use of a sample spinner to reduce the errors of particle counting statistics. The certified values include the lattice parameters, 12 relative intensity values from 25 to 145 degrees 2Θ, and absolute variation in intensity. The certification of the last of these parameters allows for comparison of absolute diffraction intensity from equipment at differing locations.

The round robin study indicated only that a problem existed with regards to variation in instrument sensitivity. With the exception of groupings in performance by manufacturer, it indicated little as to its origins. The certified relative intensity data of SRM 1976 were collected on a Philips[1] diffractometer of conventional optical layout which included a graphite diffracted beam monochromator (Cline, 1992). This "reference" machine was determined to be in "correct" alignment with respect to the results of the Jenkins, Schreiner & Dismore (1992) round robin. However, due to a lack of understanding of the mechanisms responsible for the effects observed, this machine could not be determined to be any more suited as a reference than any other properly aligned, conventional x-ray diffractometer. Early experiments found no dependence of instrument sensitivity on the size of the incident and receiving slits, or the presence or absence of soller slits, either incident and receiving.

The use of SRM 1976 for evaluation of instrument sensitivity entails the collection of data on the test instrument in a manner analogous to that used in the certification. The uniformity of the SRM material with respect to size and strain broadening (minimal) permits the use of peak heights. They were included in the certification. Evaluations performed with the use of peak heights will reflect the lower precision of this type of intensity measurement. Certified integrated intensities were measured using the Siemens version of the NBS*Quant (Hubbard, Robbins & Snyder, 1983) algorithm. The use of this algorithm is not required, but a credible method of determining a background subtracted integrated intensity measurement should be employed. Graphical evaluation of the performance of the test instrument is accomplished by plotting the ratio of the 12 relative intensities measured from the test instrument to the certified values as a function of 2Θ angle. Such a plot is shown in figure 1.

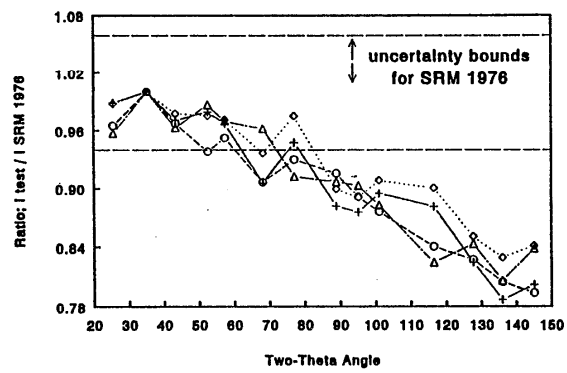

Figure 1. Graphical presentation of test machine results from four samples of SRM 1976.

The upper and lower bounds displayed in figure 1 represent the coverage of 90% confidence, 95% coverage tolerance interval for the certified relative intensity data. Tolerance intervals were used due to the expected heterogeneity of the levels of preferred orientation and thus the intensity values measured; no true mean for the population as a whole exists. The preferred orientation is expected to uniformly affect all measurements, thus data analysis assumed one true relative variance (Cline, Schiller & Jenkins, 1991).

Interpretation of the plot may require some scientific judgement on the part of the operator. Patternless scatter within the error bounds indicate the test machine performs in a manner analogous to the reference machine and needs no correction. If the scatter of the data is such that more than one peak falls outside the error bounds, a lack of precision on the part of the user measurements is indicated. Patterns observed in the data, even within the error bounds, indicate that a correction may be necessary. Procedures to test this hypothesis can be found in the appendix of the SRM 1976 certificate (Cline, 1992). The data of figure 1 clearly indicate that a correction curve should be applied to intensity data collected from this machine.

[1]Certain commercial equipment, instruments, or materials are identified in this paper in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation by NIST, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

During a realignment of the test machine of figure 1, it was determined that slight changes in the angle of the graphite monochromator crystal would cause changes in instrument sensitivity of up to 20%. The machine was a second Philips diffractometer similar to the reference, with the exception of being equipped with an automatic sample changer as opposed to a sample spinner. The effect occurred through a rocking angle of the graphite crystal within which the intensity of the (104) reflection (35.1° 2Θ) from an SRM 1976 sample did not vary significantly. The tuning of the monochromator was performed to minimize contributions from the Cu Kβ and W L lines. Alignment of the crystal to minimize this spectral contamination resulted in the calibration curves of figure 1. The test machine could be made to perform identically to the reference machine with an appropriate setting of the monochromator crystal. This observation indicates the slight variations in the graphite monochromator angle are responsible for at least part of the effects observed in the Jenkins & Schreiner round robin. A more exhaustive study of the causes of variation in instrument sensitivity is underway utilizing a survey of selected equipment of different manufacturing origin (Cline, Schiller & Jenkins, 1992).

Quantitative analysis SRMs

Quantitative analysis SRMs are powders selected to offer a diffraction intensity as close to the "ideal" as possible. They are of high phase purity, small crystallite size, and of isometric particle morphology. They may be specific to given systems, as in the case of SRMs 1878 and 1879, which are designed for quantification of quartz and cristobalite, respectively, in airborne dusts. Alternatively, SRM 674a consists of a set of five powders of varying absorptivity: α Al_2O_3, $\mu=126cm^{-1}$; ZnO, $\mu=279cm^{-1}$; TiO_2, $\mu=536cm^{-1}$; Cr_2O_3, $\mu=912^{-1}$; and CeO_2, $\mu=2203cm^{-1}$ (absorptivities are for Cu Kα radiation). This selection of materials allows for the matching of the absorptivity of the standard to that of the unknown, minimizing the effects of microabsorption.

Recently SRM 676 was certified. It consists of an α alumina powder designed for measurement of I/I_c values. The concept of the I/I_c as a constant relating a material's diffraction intensity to that of α alumina, corundum, was proposed by Visser & deWolf (1964). It is a specific case of the more general Reference Intensity Ratio (RIR) later proposed by Chung (1974 a,b). The I/I_c concept has been subsequently adopted by the ICDD and is one of the material constants included in the PDF2 database. The RIR of any two phases can be determined from the ratio of their respective I/I_c values. Thus the listing of these values in the database allows for quantitative information to be gained in a more routine manner.

The selection of the alumina powder for SRM 676 involved a study of the dependence of diffraction intensity on specific powder characteristics (Cline & Von Dreele, 1992). The first of these, obviously, is phase purity; a basic problem surfaces immediately for quantitative XRD SRMs based on commercial powders. The ceramics industry requires active powders for use in the manufacture of sintered ware. These powders are generally in the tenths of a micron size range and are of a high surface area. The resultant line broadening is undesirable, but cannot be considered a major factor in selection of quantitative analysis SRMs; a credible intensity measurement will consist of an integrated intensity. The high surface area is detrimental due to the potential increase in the mass fraction of an "amorphous" non-diffracting, surface phase. This surface phase, while possessing some degree of order, does not contribute to Bragg diffraction from the bulk crystal structure. The exact amount of the impurity phase is dependent on the lattice energy of the bulk crystal structure, the specific surface of the material, and its processing history.

The consideration of surface phase contamination is particularly problematic for aluminum oxide powders. Commercial alumina powders are products of the Bayer process wherein, initially, an aluminum hydroxide precursor is chemically generated. This precursor material is subsequently calcined through a series of well known transition aluminas until the high temperature α phase is reached (Wefers & Bell, 1972). Highly active, sinterable, alumina powders are often incompletely calcined and are phase contaminated by several of the transition aluminas. Complete conversion to the α alumina requires a long duration calcination to at least 1200C. Powders produced by this process can not be considered ideal for XRD purposes either due to their large grains, state of aggregation, and platy crystal habit. Early experiments indicated that a prerequisite for precise I/I_c measurements are fully deaggregated powders. Extensive experiments evaluated four highly calcined, deaggregated, phase pure powders of relatively small crystallite (particle) size, for suitability as this SRM material.

The experimental design considered the effects of extinction, the "amorphous" non-diffracting surface layer, and preferred orientation. The first two of these effects will manifest themselves in terms of a reduction in diffraction intensity. The isolation of the material offering the maximum diffraction intensity was accomplished by first spray drying mixtures of the test materials and silicon, SRM 640b, to eliminate the effects of preferred orientation (Cline & Snyder, 1983). An I/I_c determination of SRM 640b relative to the test

materials was performed via Rietveld refinements of XRD data collected from these and conventionally prepared specimens. Equipment consisted of a Siemens D500 diffractometer equipped with a focusing Ge incident beam monochromator, sample spinner, and a position sensitive detector. The conventionally prepared specimens provided data on the levels of preferred orientation via refinements of the March-Dollase parameter (Dollase, 1986). Extinction effects were measured using the Sabine model, (Sabine, 1985; Sabine, Von Dreele & Jorgensen, 1988) in conjunction with Rietveld analysis of time-of-flight (TOF) neutron measurements. Additional measurements included particle size via laser scattering, specific surface measurements, and examination with a scanning electron microscope.

Results from these four powders as well as the final SRM 676 material are shown in table 1. A low I/I_c value was obtained from Linde C which exhibited no extinction but had the largest surface area of the four test materials. This may indicate that the effect of its presumably higher amorphous content was offset by the extinction effects of the other candidates. However, this conclusion is not supported by the observation that the Sumitomo material exhibited considerable extinction and also displayed a favorable I/I_c value. This may indicate an as yet undetermined variable is operative or that the magnitude of the effects is below the measurement precision. The refined March-Dollase parameters, which equals one for a randomly oriented powder, indicated the Linde C material did not display preferred orientation. The SRM itself was prepared from a second lot of the Linde C product. SRM 676 was certified with respect to lattice parameters and seven relative intensity values from 24 to 78 degrees 2Θ. The certification of the relative intensity values allows the user to measure any one of the eight lines for an I/I_c determination (use of more than one line is recommended). The machine used for collection of the certified relative intensity data was the "test" machine discussed in the section concerning SRM 1976. In addition to an adjustment of the reported values, the reported uncertainty also had to reflect the measurement uncertainty of SRM 1976 leading to the calibration curve (figure 1). This was done using the statistical technique of bootstrapping. Figure 2 indicates the dramatic increase in the uncertainty from the 30 certification measurements of the (024) reflection of SRM 676 with the incorporation of the uncertainty of test machine measurements of SRM 1976. The three vertical lines represent the size that a 95% confidence interval for the mean would be when different sources of error are incorporated. The smallest (leftmost) interval includes only the variability in the 30 measurements of SRM 676. In the middle interval, the uncertainty in the calibration curve has been included as well. Finally, the third interval incorporates the uncertainty in the certified values of SRM 1976, as well as the uncertainty in the calibration curve and the variability of the measurements of SRM 676.

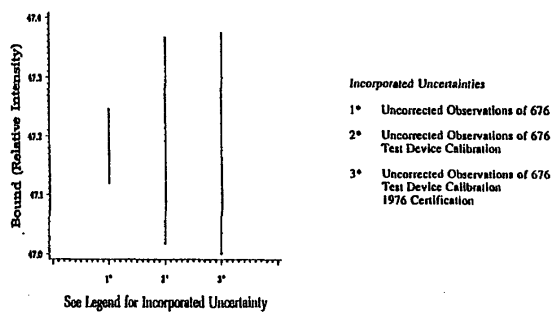

Figure 2. Comparison of 95% confidence intervals of SRM 676 certification data when different sources of error are included.

These data indicate that the uncertainty in relative intensity values, once converted to an interlaboratory scale via SRM 1976, is due as much to the uncertainty in the conversion as to the variability in the uncorrected

Table 1. Results from measurements of SRM 676 candidate materials

Material or Supplier	Mean Particle Size, μm Laser	Refined Domain Size, μm TOF data	BET Surface Area, m^2/gr	I/I_c Values for Si, SRM 640b	March - Dollase coef.
Linde C	1.5	---	3.34	4.105	.998
IBM	3.3	1.4	1.27	4.173	.886
Sumotomo	4.3	2.5	.93	4.097	.818
Alcan	4.0	2.7	1.07	4.176	.767
SRM 676	1.4	---	4.98	4.104	.998

measurements themselves. This suggests that users of SRM 1976 need to incorporate the uncertainty of this correction into the total of their measurements. The bootstrapping approach used here is not practical for user at other laboratories, but a simpler approximation is being developed at NIST and will be described in an upcoming publication (Cline, Schiller & Jenkins, 1992).

Another new quantitative analysis SRM, which is presently being certified, is specific to analysis of silicon nitride (Cline & Von Dreele, 1992). The ceramics industry is interested in silicon nitride due to its desirable high temperature properties. Ubiquitous to processing this material is the transformation of the starting low temperature, α phase to the high temperature, β phase. Approximately 90% of typical commercial powder designed for the production of sintered ware will consist of the α phase, with the remainder being amorphous material and the β phase. Upon sintering, the α phase transforms into the β, resulting in an interlocking microstructure of acicular β grains. This interlocking microstructure, along with the intrinsic properties of the β phase of Si_3N_4, are responsible for the desired high temperature performance and fracture toughness of this material.
The α to β ratio of both starting material and the subsequent sintered ware is known to affect properties and is the primary issue addressed by this SRM. However, after discussions with industrial representatives, it became apparent that the ability to analyze for amorphous content was also desired. The SRM itself will consist of two powders, one high in α content, the other high in β. Each is to be certified with respect to α/β ratio and the amorphous content and thus will be suitable for use as a spiking material for user measurements.

The analysis of amorphous content can be performed with the assumption that a spiking phase is 100% crystalline. Samples of the commercial "α" material selected as the SRM powder were spiked with various concentrations of a phase pure β whisker material. XRD samples were spray dried. Rietveld refinements of XRD and TOF data allowed the refinement of a histogram scale factor and the unit cell fraction of the two phases. The use of the Rietveld method circumvents any difficulty caused by the complete interference of the β phase's diffraction profiles with those of the α. The failure of measured unit cell fractions to follow the expected dependence on the concentration of the spiking phase can be entirely attributed to material in the analyte which is not contributing to Bragg diffraction from either phase, defined in this case as the "amorphous phase." Equations are derived wherein a measured unit cell fraction and the mole fraction of the spikant are related to the mole fractions of the α and β phases in the analyte:

$$S_\alpha = \frac{\frac{x_\alpha}{Z_\alpha} f_\alpha}{f_\alpha\left(\frac{x_\alpha}{Z_\alpha} + \frac{x_\beta}{Z_\beta}\right) + \frac{f_\beta}{Z_\beta}}$$

a similar equation may be written for S_β. Also:

$$x_\alpha + x_\beta + x_A = 1$$

where:

f_α = mole fraction of "pure" α phase
f_β = mole fraction of β phase

contents of "pure" α phase; unknowns

x_α = mole fraction α phase
x_β = mole fraction β phase
x_A = mole fraction amorphous phase

$Z_\alpha = 4$ S_α = unit cell fraction of α phase
$Z_\beta = 2$ S_β = unit cell fraction of β phase

Rearranging the first equation and substituting $1-S_\alpha$ for S_β gives:

$$\frac{(1-f_\alpha)}{f_\alpha} = x_\alpha \frac{(1-S_\alpha)}{2S_\alpha} - x_\beta$$

Graphical presentation of this linear relation for the XRD and TOF data is shown in figure 3. The slope of this plot indicates the α content while the y intercept indicates the β content and the difference between the two indicates the amount of the amorphous phase. The XRD data indicate a higher amorphous content than the TOF data which is believed to be caused by a humidity induced atmospheric reaction with one or both phases. This reaction can be assumed to affect the specimens of Gaithersburg, MD more than those of Los Alamos NM. These data indicate this silicon nitride powder consists of 93.8% α phase, 3.4% β phase, and 2.8% amorphous phase. This technique can be extended to the measurement of the amorphous or impurity level of any material relative to a second material which can be considered phase pure. The "impurity level" can be considered as the amount of any material which is not contributing to Bragg diffraction from either phase. The extension of this technique to other quantitative SRMs is a topic of current investigation.

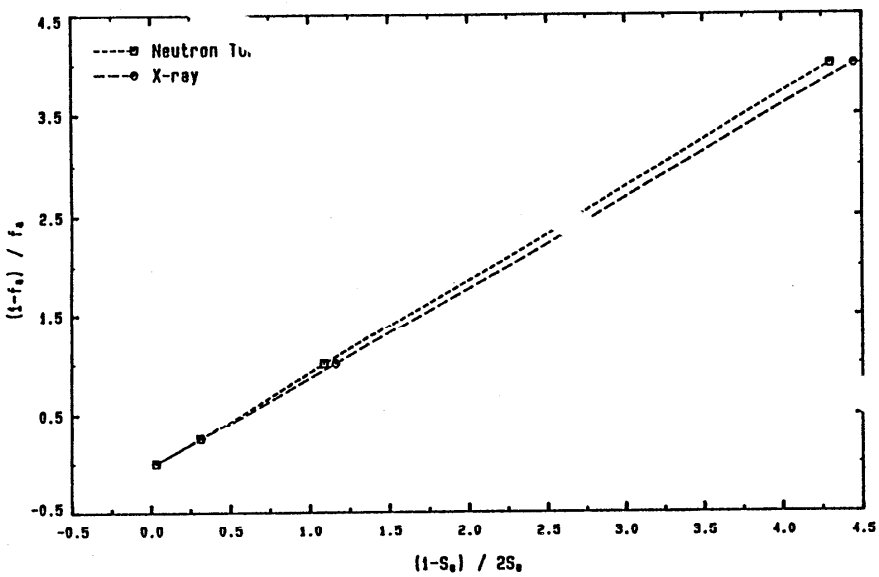

Figure 3. Graphical analysis of TOF and XRD data for the silicon nitride SRM powder of high α phase content.

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