รายงานวิจัยฉบับสมบูรณ์

การศึกษาค่ากำลังดัดขวางและการวิเคราะห์การเลี้ยวเบนรังสีเอกซ์ของเซรามิคลิวไซต์ปริมาณสูงภายใต้ภาวะการเย็นตัวลงต่างกัน

Flexural Strength Evaluation and X-ray Diffraction Analysis of High Leucite Ceramic under Various Cooling Conditions.

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รายงานวิจัยฉบับสมบูรณ์

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"การศึกษาค่ากำลังดัดขวางและการวิเคราะห์การเลี้ยวเบนรังสีเอกซ์ของเซรามิคลิวไซต์ปริมาณสูงภายใต้ภาวะการเย็นตัวลงต่างกัน"

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เซรามิกที่มีลูไซต์ปริมาณสูง สามารถนำมาขึ้นรูป เป็นครอบฟัน อุดฝัง/อุดครอบ และวีเนียร์ ด้วยกรรมวิธีอัดฉีดเข้าเบ้า โดยมีผลึกลูไซต์ช่วยเพิ่มความต้านทานการแตกหักให้กับแก้วแมทริก การทดลองนี้มีจุดประสงค์เพื่อศึกษาผลของการเย็นตัวลงที่มีต่อกำลังดัดขวางของแท่งเซรามิก (IPS-Empress, Ivoclar) เตรียมแท่งเซรามิก 60 แท่ง ขนาด 2.0 x 1.5 x 25 มม. แบ่งเป็น 3 กลุ่ม ๆ ละ 20 แท่ง โดยนำแท่งเรซิน (GC resin pattern) มาทำเบ้า โดยนำเบ้าพร้อมอินกอตชนิดที่สอง (T2) เข้าเผาในเตาเผาก่อน ที่อุณหภูมิ 850° ซ จากนั้นย้ายเบ้ามาเผาต่อที่เตาเผาเอมเพรส จนถึงอุณหภูมิ 1180° ซ ตามคำแนะนำบริษัท เมื่อเผาครบวงจร จึงปล่อยให้เบ้ามีการเย็นตัวลง 3 วิธี คือ กลุ่มที่ 1) นำเบ้าออกจากเตาทันที นำมาวางไว้ในที่แห้ง ณ อุณหภูมิห้อง 23° ซ กลุ่มที่ 2) นำเบ้าออกจากเตาทันที โดยจุ่มลงในน้ำที่อุณหภูมิ 23° ซ และ กลุ่มที่ 3) ปล่อยให้เบ้าเย็นตัวลงอย่างช้า ๆ ในเตาเผา จนถึงอุณหภูมิ 23° ซ นำแท่งเซรามิกทั้งหมดมาทดสอบค่ากำลังดัดขวางแบบ 3 จุด ด้วยเครื่องทดสอบสากล ความเร็วตัดขวาง 0.2 มม./นาที นำข้อมูลมาเปรียบเทียบโดยใช้สถิติวิเคราะห์ความแปรปรวน และการทดสอบทูกีย์ ที่ระดับความเชื่อมั่น 95 และวัดปริมาณลูไซต์ในแต่ละกลุ่มด้วยกรรมวิธีวิเคราะห์การเลี้ยวเบนรังสีเอกซ์เชิงปริมาณ โดยมีอลูมินาเป็นสารมาตรฐาน ค่ากำลังดัดขวาง (x ± SD, เมกะปาสคาล) และปริมาณลูไซต์ (%) ดังนี้ กลุ่มที่ 1) 69.97 ± 19.52,12.6 กลุ่มที่ 2) 63.88 ± 15.77, 8.7 และกลุ่มที่ 3) 85.76 ± 12.84, 16.6 แท่งเซรามิกที่ถูกปล่อยให้เย็นตัวลงอย่างช้า จะให้ค่ากำลังดัดขวางสูงสุดและมีปริมาณลูไซต์มากที่สุดด้วย แต่กลุ่ม 1 และ 2 นั้น ไม่มีความแตกต่างทางสถิติ จึงสรุปได้ว่า ภาวะการเย็นตัวลงที่ต่างกัน มีผลต่อค่ากำลังดัดขวางของวัสดุทดสอบ

ข้อเสนอแนะ: เพื่อให้เข้าใจชัดเจนถึงการที่ผลึกลูไซต์มีผลต่อความต้านทานการแตกหักของชิ้นงานเซรามิก ในอนาคต ควรศึกษาถึงการกระจายของผลึกลูไซต์ในแก้วแมทริกในขณะเย็นตัวลง และขนาดของผลึกลูไซต์

คำหลัก: เซรามิคที่มีลูไซต์ปริมาณสูง ค่ากำลังดัดขวาง การวิเคราะห์การเลี้ยวเบนรังสีเอกซ์ ระบบการฉีดเข้าเบ้า

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Leucite reinforced ceramic fabricated using an injection molding system can produce a variety of ceramic restorations such as crowns, inlays/onlays and veneers. The leucite crystals improve fracture resistance of the glass matrix. This study investigated the effect of cooling conditions on the flexural strength of high-leucite content ceramic bars (IPS-Empress, Ivoclar). 60 bars (2.0 x 1.5 x 25 mm) were fabricated (20 bars per group). Resin bars (GC resin) were invested as per a lost wax technique. Molds and ingots (T2) were first heated in a furnace to 850° C and then transferred to an IPS-Empress furnace and heated to 1180° C, following their manufacturers' recommendations. When the heating cycle was completed, molds were subjected to 3 different cooling conditions. Group 1) molds were immediately removed and placed in a dry environment at room temperature (23° C); Group 2) molds were immediately removed and placed in water at 23 ° C; Group 3) molds were allowed to cool slowly inside the furnace to 23° C. Bars were tested in three-point flexure on a universal testing machine (crosshead speed 0.2 mm/min). ANOVA and Tukey statistical analyses ($p<0.05$) were performed on the data. The leucite content of each group was measured by quantitative x-ray diffraction using alumina standards. Flexural strengths ($X \pm SD$, MPa) and leucite content (%) are Group 1) 69.97 ± 19.52, 12.6; Group 2) 63.88 ± 15.77, 8.7; Group 3) 85.76 ± 12.84, 16.6 . Bars were allowed to cool slowly gave the highest flexural strength and a maximum leucite content. There is no significant difference ($p>0.05$) between Group 1 and 2. The cooling condition has an effect on flexural strength of tested materials.

Further Study: In order to fully understand how leucite crystals could improve the fracture resistance of a ceramic restoration. The distribution of leucite crystals in a glass matrix upon cooling, in conjunction with these crystal sizes should be further investigated.

Key words: high- leucite ceramic, flexural strength, X-ray diffraction analysis, an injection molding system.

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INTRODUCTION

Currently in routine dental practice, many patients demand esthetic restorations (Mormann et al., 1989). Tooth-colored restorative materials have been introduced to replace missing tooth structure (Craig, 1993). With respect to anterior teeth, composite resins and porcelain laminate veneer restorations are successfully used for the treatments of discolored, malformed or mal-positioned teeth and diastema (Horn, 1993; Calamia, 1985; Calamia et al., 1987). Veneers provide excellent esthetics and conserve tooth structures (Yen et al., 1993). Esthetics for posterior restorations, therefor, is taken into consideration because of patients' desires.

Dental silver amalgam is commonly used in operative dentistry especially in the occlusal load-bearing area of posterior teeth. The replacement of amalgam restorations with tooth-colored restorations is increasing (Roulet et al., 1991). Composite resin and porcelain restorations are, therefore, available to achieve a suitable esthetic appearance in either anterior or posterior teeth (Mormann et al., 1989).

Composite resin materials can be made directly by dentists or indirectly processed from an impression in a

dental laboratory. The clinical longevity of composite resin is still uncertain (Mormann et al., 1989; Craig, 1993). The difference in modulus of elasticity and coefficient of thermal expansion between composite resin and enamel are believed to be causative factors for marginal leakage or segregation (Roulet et al., 1984; Mormann et al., 1989). Composite resin tends to have volumetric polymerization shrinkage of approximately 2.0% to 3.2% (Lutz et al., 1986). Wear and loss of marginal integrity are still unsolved problems (Roulet, 1987; Craig, 1993).

Ceramics, in contrast to composite resins, have physical and chemical properties more similar to enamel (Adair and Grossman, 1984; Hobo and Iwata, 1985a; Hobo and Iwata, 1985b). Ceramics are also generally accepted to be very biocompatible with oral tissues. Light absorption and light scattering behavior provide the ability to mimic the appearance of natural teeth. Ceramics are therefore used mostly for esthetic fixed restorations (Nathanson, 1987). The artistic skill of a dental technician is often required to create a life-like appearance of the ceramic restoration.

Furthermore, ceramics, by nature, are extremely brittle and easily fracture under stress. In ceramo-metal substructure which is designed to strengthen the

restoration. The metal substructure prevents bending of veneering porcelain and subsequently inhibits crack propagation.

There are certain limitations, however, to the use of porcelain. Mechanical failure of ceramic materials is almost completely controlled by brittle fracture. The brittle behavior, combinded with the presence of surface flaws, resulted in relatively low ceramic strengths and has limited use of ceramic materials.

The production of a satisfactory porcelain restoration requires careful attention to the nature of the porcelain. Porcelain contains feldspar, quartz, clay, color frits, and fluxes in different proportions, depending upon manufacturers' formulas. Even though porcelain contains glass and crystalline phases, the glass phase is generally prominent. Porcelain tends to shrinkage during sintering in the furnace. The volumetric shrinkage can be compensated by over-building the pre-baked porcelain in combination with a good condensation of the porcelain.

Ceramic restorations can be fabricated in different manners such as: 1) conventional porcelain sintered on a refractory die technique; 2) a castable ceramic onlay/inlay; 3) injection molded ceramic system; 4) CAD/CAM (computer assisted design/ computer assisted

milling) and 5) slip-cast echnique; and 6)porcelain milling system (Giordano, 1996).

Ceramic onlay/inlay restorations are effectively bonded to enamel using composite resin as a luting medium. The enamel surface is etched with phosphoric acid in order to provide a strong micro-mechanical bond between resin and enamel (Bounocore, 1963). Ceramics can also be successfully etched with some types of hydrofluoric acid-based etchants to enhance the bond strength (Simonsen and Calamia, 1993; Calamia et al., 1985; Calamia, 1985; Hsu et al., 1985; Calamia et al., 1987; Chan et al., 1987; Lacy et al., 1987; Nathanson, 1987; Kanchanatawewat and Stannard, 1989).

When thin etched porcelain (which is very fragile and easily fractured) is bonded to etched enamel, the tooth can prevent the porcelain from distorting as well as inhibit crack propagation in porcelain. This principle is well accepted and allows porcelain to be successfully used for laminate veneers or onlay/inlay restorations (Nathanson, 1987).

All-ceramic restorations are thought to provide better esthetics compared to the porcelain-fused-to-metal restorations (Scherrer et al., 1994). Metal substructures, not only interfere with the light scattering behavior of the restorations but also have an

effect on biocompatibility with the oral tissue. The release of metallic elements from dental alloys in an oral cavity is a potential health problem to the dental patient. In sufficient concentrations, metals are known to cause toxic, inflammatory, allergic, or mutagenic reactions (Wataha et al., 1995).

However, the brittle nature of ceramics and their poor fracture resistance play a major role in the longevity of such restorations (McLean, 1983; White et al., 1994). Several techniques have been introduced to strengthening dental ceramics. When crystalline materials such as leucite (K_2O-Al_2O_3-$4SiO_2$), alumina (Al_2O_3) or tetrasilicic fluoromica ($K_2Mg_5Si_8O_{20}F_4$) are added to glass matrix, the glass is toughened and strengthened because cracks can not penetrate the reinforcing components (Anusavice, 1996). These reinforcing components provide fracture resistance and improve strength because of the generation of compressive stresses that surround the crystals. This technique has been applied in the development of an injection-molded system (IPS-Empress) by the department of fixed and removable prosthodontics and dental materials at the University of Zurich, Switzerland in conjunction with Ivolclar, Schaan, Liechtenstein in 1983.

IPS-Empress system is designed for the fabrication of single units, onlays/inlays and veneers. (Dong et al., 1992) This ceramic is a feldspar-based ceramic material having the following composition in wt %: 63 SiO_2, 17.7 Al_2O_3, 11.2 K_2O, 4.6 Na_2O, 0.6 B_2O_3, 0.4 CeO_2, 1.6 CaO, 0.7 BaO, 0.2 TiO_2. The reinforcing components of this glass-ceramic composed of leucite crystals have the particle sizes ranging from 3 to 5 μm in the glass matrix. (Seghi and Sorensen, 1995)

This injection-molded system required a special furnace (Empress EP 500). The furnace consisted of an enlarged heating chamber, a pneumatic pressure system, a reducing valve, and a manometer to control the pressure. An inductive displacement transducer is mounted on top of the furnace and is connected to the pneumatic plunger. When the programmed temperature is reached, the heated ingot is pressed into the mold under a pressure of 5 bars. The flexural strength of an heated ingot increases approximately 19%. (Dong et al., 1992)

The word "leucite" is derived from Greek, which means "white". In general, leucites are trapzohedral crystals at which in low temperature are pseudomorphs of the high-temperature form. Leucite crystals, by nature are luster vitreous to dull, having white or gray color. Leucite (K_2O-Al_2O_3-$4SiO_2$) is a mineral formed the

incongruent melting of feldspar (K_2O-Al_2O_3-$6SiO_2$) and composed of 21.5% K_2O, 23.5% Al_2O_3, and 55.0% SiO_2.

Leucite crystals also have linear coefficient of thermal expansion higher than feldspar glass matrix. When heated leucite contained ceramic is cooled, the compression zones around those crystals are subjected to yield crack propagation thus increase the overall strength (Kanchanatawewat et al., 1997). Heating conditions such as repeating firings or certain heat treatments can possibly alter the thermal expansion behavior of crystalline phase leucite and such changes in expansion could induce cracking of porcelain (Märckert et al., 1986).

Dong et al. showed that the injection molded procedure provided better dispersion of the leucite crystals in the glassy matrix, subsequently enhanced the flexural strength (Dong et al., 1992). The amount of leucite content is thought to be responsible to the strength of the ceramic (Mackert and Russell, 1996).

While the longevity clinical perfomance of all-ceramic restorations depends on several factors, the relative strength of these materials is of significant interest. There are several test methods for the evaluation of the strength of porcelain. Because of the clinical problem in all-ceramic restorations is associted

with the direct tension of brittle materials, a bending test is the most widely used for testing ceramics and generally considered the most satisfactory means of assessing strength. The three-point beam test is simple and reliable procedure. It is extremely sensitive to surface conditions (Edwards et al., 1983; Seghi et al., 1990; and Seghi and Sorensen, 1995) . The authors recommended that this test is ideal for the evaluation of brittle materials especially dental ceramics. However, the porcelain specimens were relatively difficult to fabricate. The International Standards Organization (ISO-6872, 1995 currently supports the use of the simple three-point bending test for the evaluation of strength of dental porcelain (Seghi et al., 1990).

The understanding of strengthening mechanism of leucite enriched ceramic is important. The aim of this study was to evaluate the flexural strength and to determine the amount of leucite dispersed in the IPS-Empress ceramic under three different cooling conditions.

MATERIALS AND METHODS

A. Preparation of IPS-Empress bars

The use of an injection molded system (IPS-Empress, Ivoclar, Bendererstrasse 2, FL-9494 Schaan, Liechtenstein) has been introduced to fabricate all-ceramic restorations. Sixty auto-polymerized acrylic resin patterns (GC Pattern Resin, GC Dental Products Corp., 2-285 Toriimatu-Cho, Kasugai Aichi, Japan), having a dimension of 2.0 mm x 1.5 mm x 25 mm were made. Four resin patterns were sprued in one ring with a wax wire 3 mm in diameter and 8 mm in length and then invested into a phosphate-bonded investment as per a lost-wax technique as shown in Figure 1. A mold , a partially ceramic glass leucite ingot (T2 ingot having no color) and a plunger were heated in a burn-out furnace. The furnace temperature was raised from room temperature to 250° C, and hold for thirty minutes. The mold was heated to 850° C with a ninety minute hold and then was transferred to an IPS-Empress EP 500 furnace. The mold was then heated from 500° C to 1180° C under a pressure of 5 bars having a heat rate of 60° C per minute followed by a two minute hold according to the manufacturer's recommendations. The diagram of the basic principle of IPS-Empress is

presented in Figure 2. When the heating cycle was completed at the highest temperature, the mold was subjected to different cooling conditions.

Ninety high leucite porcelain bars were fabricated with the size of 2 mm x 1.5 mm x 25 mm, as shown in Figure 3. There were twenty bars per group. Group 1) when the heating cycle was completed, the mold was immediately removed from the furnace and placed at room temperature (23° C) in a dry environment for a fast cooling. Group 2) after the heating cycle was completed, the mold was left inside the furnace until the furnace temperature was at 23° C for a slow cooling. Group 3) when the heating cycle was completed, the mold was immediately removed from the furnace and placed in water at 23° C for a fast cooling.

After divesting, bars were then blasted (Microetcher, Danville Engineering Inc., 115A Railroad Avenue, Danville, California 94526, USA) with 100 micron glass beads (Belle de st. claire, 20600 Plummer Street, Chatsworth, California 91311-5121, USA) at sixty psi. Figure 4 shows IPS-Empress bars prior to sprue removal. Sprues were removed using a thin diamond disc (Dedeco international Inc., Long Eddy, New York 12760, USA).

B. Flexural strength evaluation

Sixty high-leucite porcelain samples having three different cooling conditions were evaluated for flexural strength using a three-point bend test, according to ASTM Standard C-1161-90, as shown in Figure 5. There were twenty samples per group. The samples were ceramic bars having the dimensions of 2 mm in width, 1.5 mm in height and 25 mm in length and tested using a universal testing instrument (Model 558 Instron Corp., 100 Royall Street, Canton, Massachusetts 02021, USA), as shown in Figure 6. A crosshead speed of 0.2 mm per minute and a load cell of 5 KN were used. The span length was 20 mm.

Means and standard deviations were calculated for all treatment groups. An analysis of variance and Tukey's HSD test with $p < 0.05$ for different treatment groups were performed, using a computer program, SPSS for windows (SPSS Inc., 444 N. Michigan Avenue, Chicago, Illinois 60611, USA).

C. Preparation of leucite standards

Natural leucite crystals (Ward's Natural Science Establishment, Rochester, NY) were crushed in a percussion mortar and ground to a fine particle with an agate mortar and pestle. Any possible iron contamination was removed by passing a strong magnet over. The leucite

particles were sieved with a 38 mm standard mesh screen. Grinding was continuously performed until all of the specimens passed through the sieve. The percent of sieved leucite particles used in the standard were 0, 25, 50, 75, 100 and the balance was silica quartz. An internal standard, Al_2O_3, was used to correct for potential absorption differences between leucite and the glass matrix. Standards were prepared by blending weighed portions of leucite and glass to yield the desired leucite weight fractions. The internal standard, Al_2O_3, was added in the amount of 25% by weight of the total mass of leucite plus glass. The standards were prepared in random sequence to avoid systematic bias resulting from preparation variables.

Diffraction patterns were recorded of the peaks in the 2θ range 29.6 to 32.3 degrees and the alumina peaks in the 2θ range 34.6 to 35.5 degrees. The patterns were recorded with a step size of 0.05 degrees and a dwell time of 6 seconds per step.

D. The quantitative x-ray diffraction analysis

After the completion of the firing circle, bars of each group were crushed in a percussion mortar and ground to a fine particle using an agate mortar and pestle. Weighed portions of each group were blended with 25 wt%

Al_2O_3, as an internal standard. Blending was accomplished by grinding the particles together with an agate mortar and pestle and subsequently sieved with a 38 mm standard mesh screen. Specimens of each group were packed onto the well of the sample holder and then analysed using an x-ray diffraction machine (model X pert, Philips Electronics N.V., Eidhoven, The Netherlands). Integrated intensities of the leucite peaks in the range 29.6 to 32.3 degrees, and integrated intensities of the alumina peaks on the range 34.6 to 35.5 degrees, were determined in the same manner as the leucite standards. The relative amount of leucite contents in each group were calculated from the calibration curve using a computer program (Quantitative Analysis Model Straight Line).

E. Fractographic analysis

After ceramic specimens were fractured, representative samples from each of the groups were also examined using a scanning electron microscope (Model XL20, Philips Electronics, N.V. Eindhoven, The Netherlands) in order to study the fractured cross-sectional surface to evaluate failure origin.

Figure 1: Resin patterns were sprued (left) and then invested with a phosphate bonded investment mold as per lost-wax technique. Mold sectioned in half.

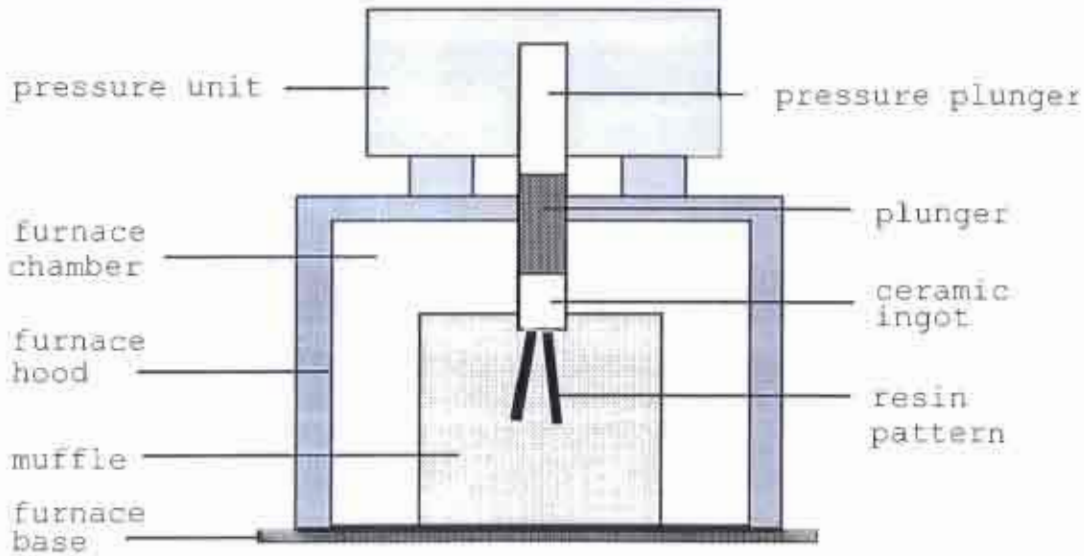

Figure 2: Diagram of the basic principle of IPS-Empress System.

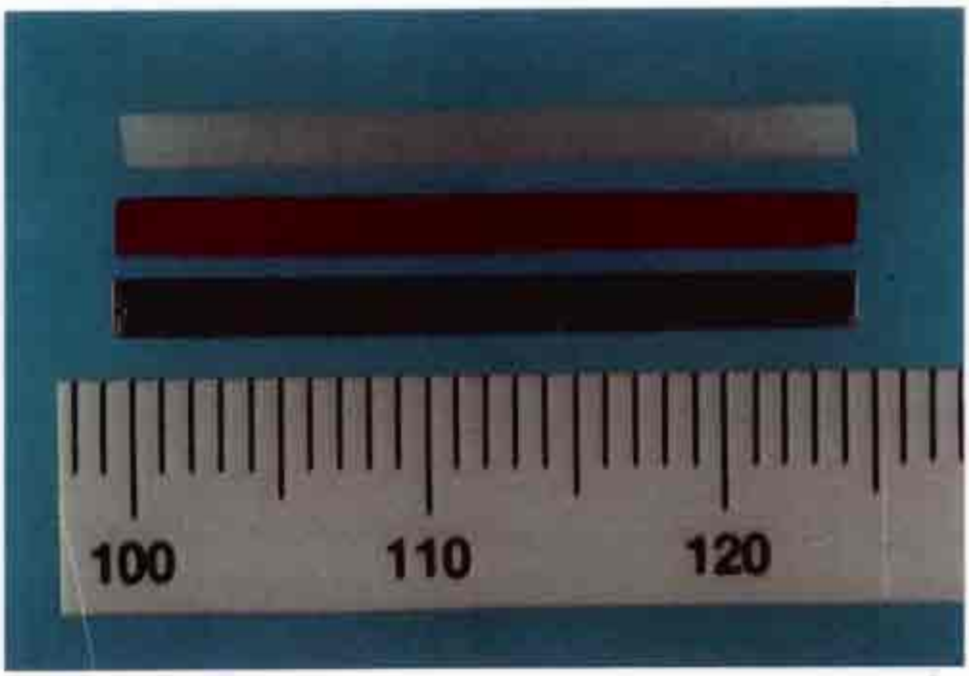

Figure 3: Example of a ceramic bar, 2 mm in width, 1.5 mm in depth and 25 mm in length, fabricated for flexural strength measurement.

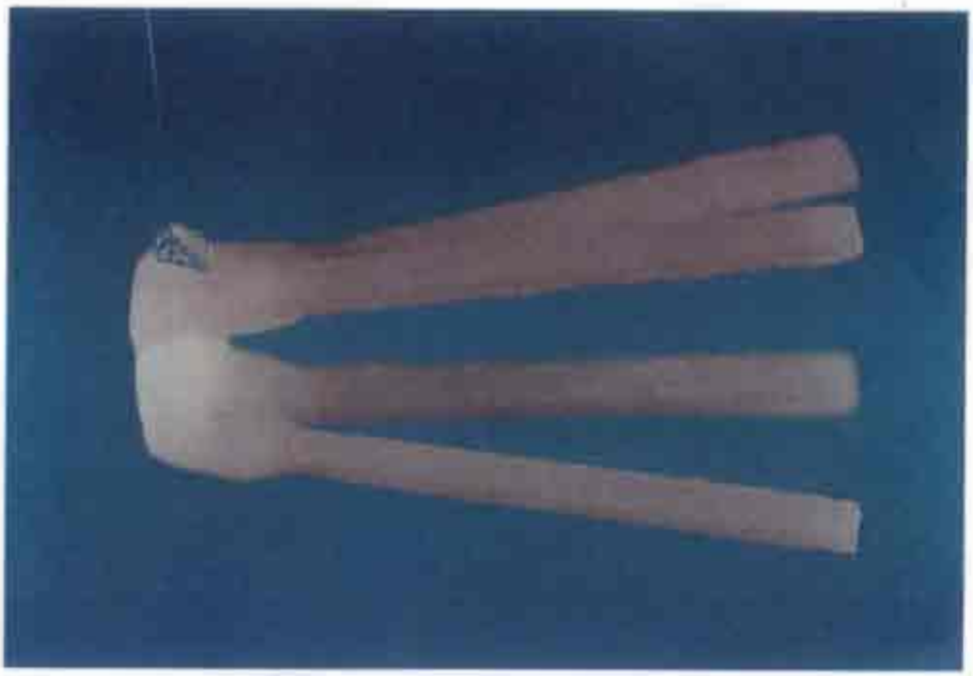

Figure 4: IPS-Empress bars prior to sprue removal.

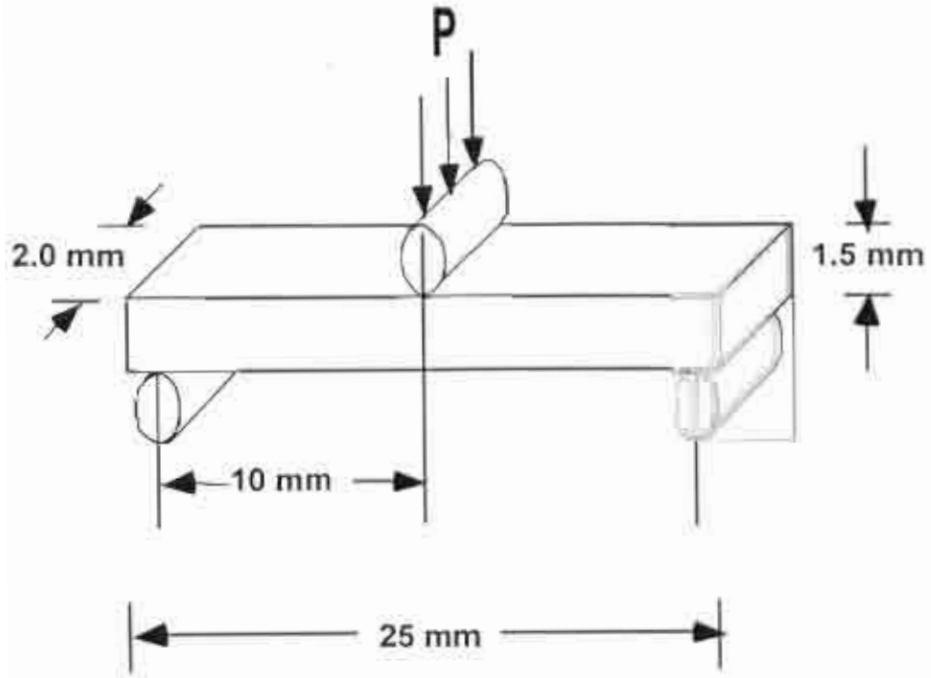

Figure 5: The Three-point fixture configuration.

Support span (L)	20 mm
Loading span	10 mm
Bearing diameter	2.0 to 2.5 mm
Width (b)	2.0 mm
Depth (d)	1.5 mm
Length (L_T)	25 mm

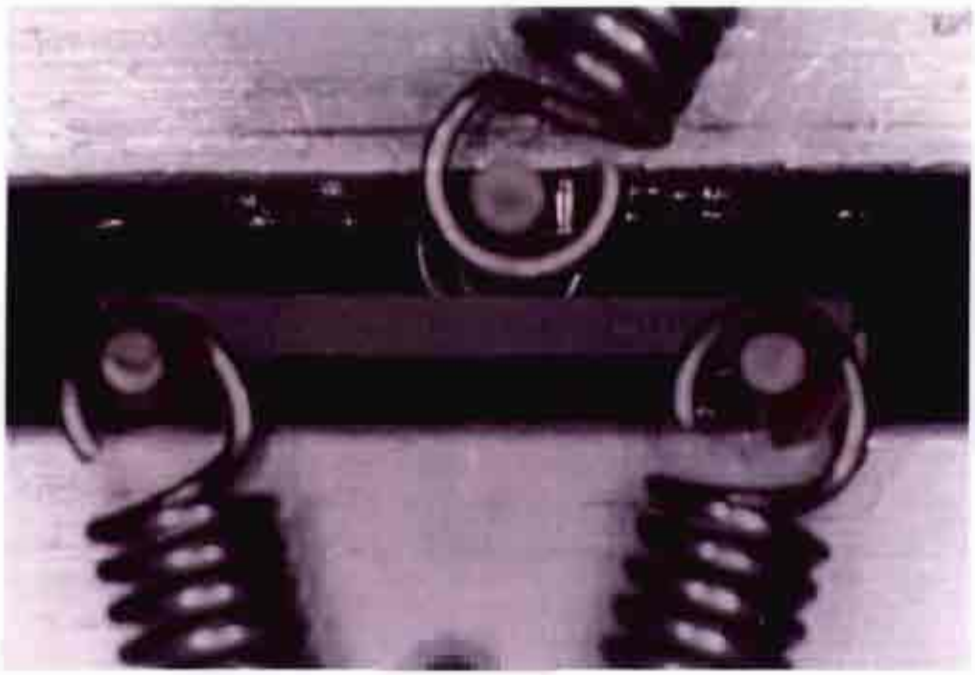

Figure 6: Bar was subjected to three-point beam test using a universal testing instrument.

RESULTS

A. Flexural strength evaluation

The flexural strength values (MPa) were calculated with the formula:

$$\frac{3PL}{2WT^2}$$

P = force recorded at fracture

L = distance between the supports (20 mm)

W = width of porcelain bar

T = thickness of porcelain bar

Equation 1. Flexural Strength Formula

Mean flexural strength values and a standard deviation of each group are presented in Table 1. The statistical analysis of variance (ANOVA) and multiple comparison testing (Tukey's test) with a significant level of $p<0.05$ were applied to the data using a computer program, SPSS for Windows (SPSS Inc. Chicago, Illinois 60611, USA), Table 2 and 3.

The mean flexural strength of Empress bars that were removed immediately from the furnace after complete firing and left in a dry environment at 23° C were 69.9667

+/- 19.5159 MPa and was not significant difference ($p < 0.05$) from the bars that were allowed to quench in water at 23° C. Empress bars that were left in the furnace after complete firing gave the highest mean flexural strength ($p < 0.05$) of 85.7598 +/- 12.8352 MPa. The Empress bars that were allowed to quench in water at 23° C showed the lowest flexural strength of 63.8804 +/- 15.7723 MPa.

B. The quantitative x-ray diffraction analysis

The quantitative x-ray diffraction analysis (Table 4) showed that high leucite ceramic which was left in the furnace after the heating cycle was completed gave the highest amount of leucite crystals (16.6%). The ceramic that was immediately removed from the furnace and was placed in water showed the lowest amount of leucite crystals (8.7%). The high leucite porcelain that was immediately removed from the furnace and was placed at room temperature in a dry environment contained 12.6% leucite crystals. The other reinforcing component such as quartz was also found mixed with all samples in an unknown proportion.

Table 1. Mean flexural strengths (MPa) of IPS-Empress bars having 3 different cooling conditions; Group 1) bars were allowed to quench in a dry environment at 23° C; Group 2) bars were allowed to quench in water at 23 ° C; and Group 3) bars were allowed to a bench cool at 23 ° C in the furnace.

Sample	Flexural Strength (MPa)		
	Group 1	Group 2	Group 3
1	90.21	37.88	75.51
2	80.86	46.79	75.65
3	49.80	63.36	73.05
4	55.81	44.70	83.48
5	61.71	53.16	92.64
6	46.88	61.04	88.67
7	45.54	53.98	70.38
8	35.37	48.97	98.69
9	65.34	61.16	74.09
10	33.75	60.15	64.63
11	87.20	61.91	99.42
12	75.85	57.86	96.69
13	80.91	70.01	103.92
14	81.27	62.63	105.17
15	81.83	91.78	73.38
16	74.88	67.48	92.36
17	81.69	67.08	74.81
18	76.89	80.94	104.92
19	98.69	90.61	81.57
20	94.86	96.11	86.16
Mean	69.97	63.88	85.76
SD	19.52	15.77	12.84

Table 2 One-Way Analysis of Variance for IPS-Empress bars having different cooling conditions; Group 1) bars were allowed to quench in a dry environment at 23° C; Group 2) bars were allowed to quench in water at 23 ° C; and Group 3) bars were allowed to bench cool at 23 ° C in the furnace.

Analysis of Variance

Source	D.F.	Sum of Squares	Mean Squares	F Ratio	F Prob.
Between Groups	2	5101.1668	2550.5834	9.6324	.0002
Within Groups	57	15093.1911	264.7928		
Total	59	20194.3579			

Table 3 The Tukey-HSD test for IPS-Empress bars having different cooling conditions; Group 1) bars were allowed to quench in a dry environment at 23° C; Group 2) bars were allowed to quench in water at 23 ° C; and Group 3) bars were allowed to bench cool at 23 ° C in the furnace.

Group	1	2	3
1			
2	NS		
3	*	*	

* = Significantly different at $p < 0.05$ using Tukey HSD test.
NS = No statistical difference.

Table 4 Quantitative x-ray diffraction analysis for IPS-Empress bars having different cooling conditions; Group 1) bars were allowed to quench in a dry environment at 23° C; Group 2) bars were allowed to quench in water at 23° C; and Group 3) bars were allowed to bench cool at 23° C in the furnace.

Group	Leucite (%)
1	12.6
2	8.7
3	16.6

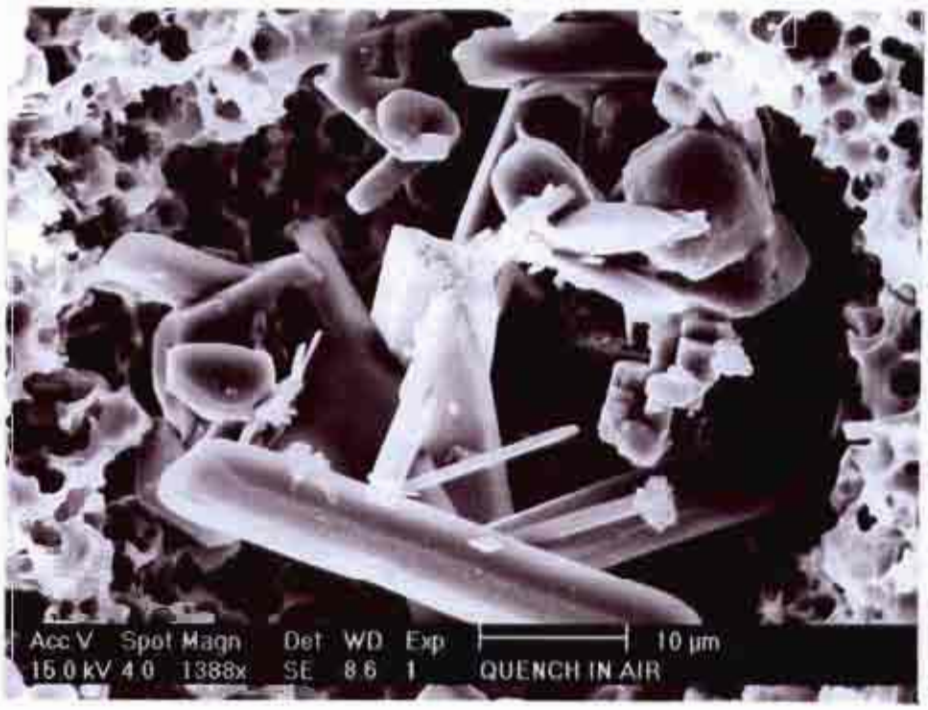

Figure 7: Scanning electron micrograph (1388x) of the IPS-Empress bar subjected to a fast cooling in air.

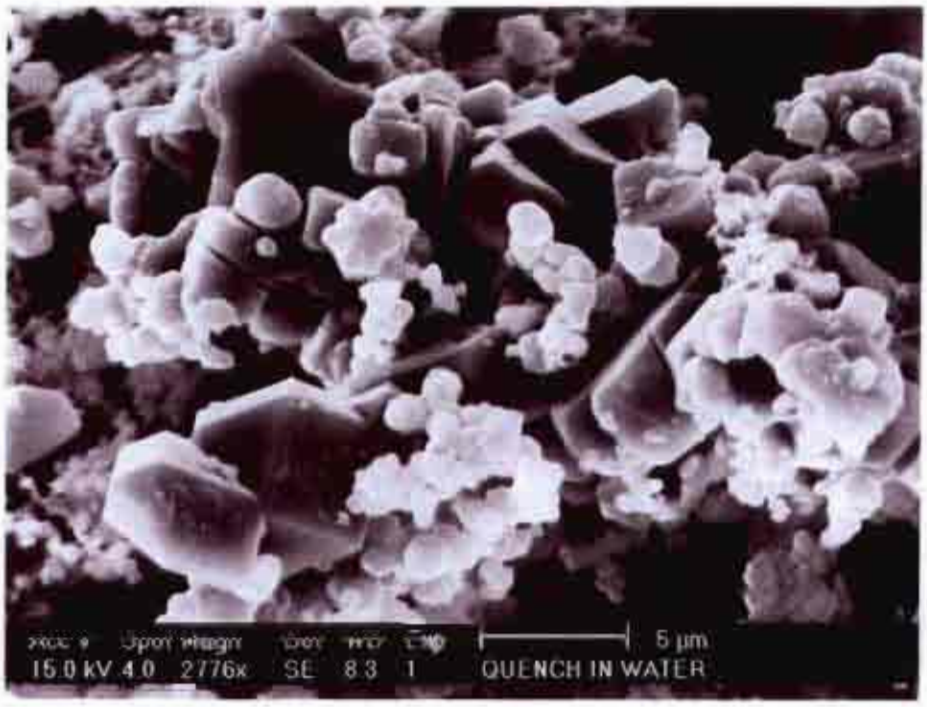

Figure 8: Scanning electron micrograph (2776x) of the IPS-Empress bar subjected to a fast cooling in water.

Figure 9: Scanning electron micrograph (1388x) of the IPS-Empress bar subjected to a slow cooling in air.

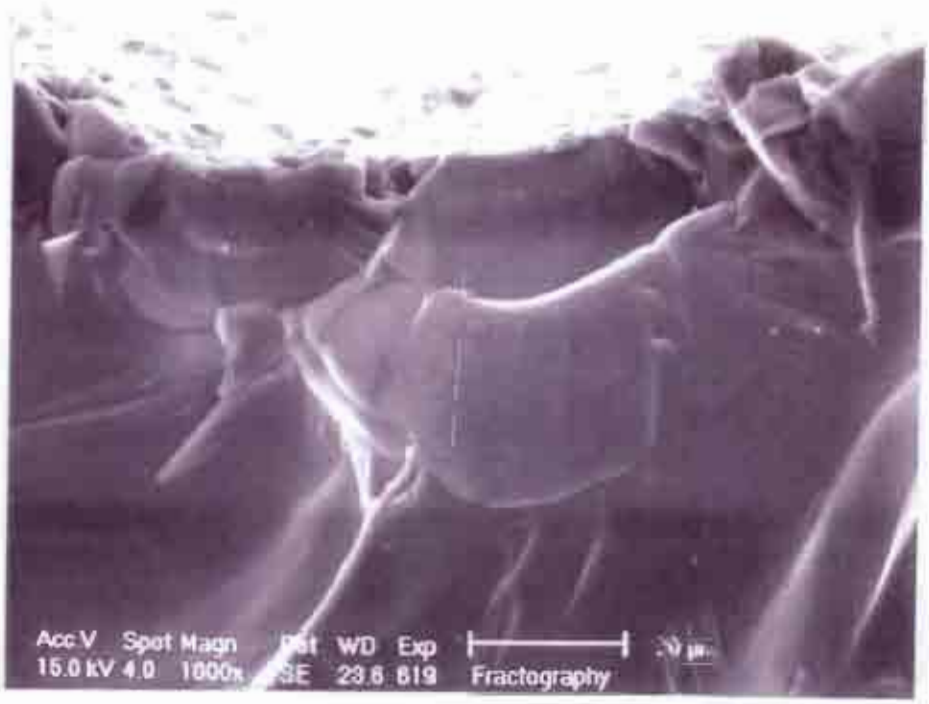

Figure 10: Fractographic analysis(1000x) of the IPS-Empress bar showed the fracture origin at the tension side

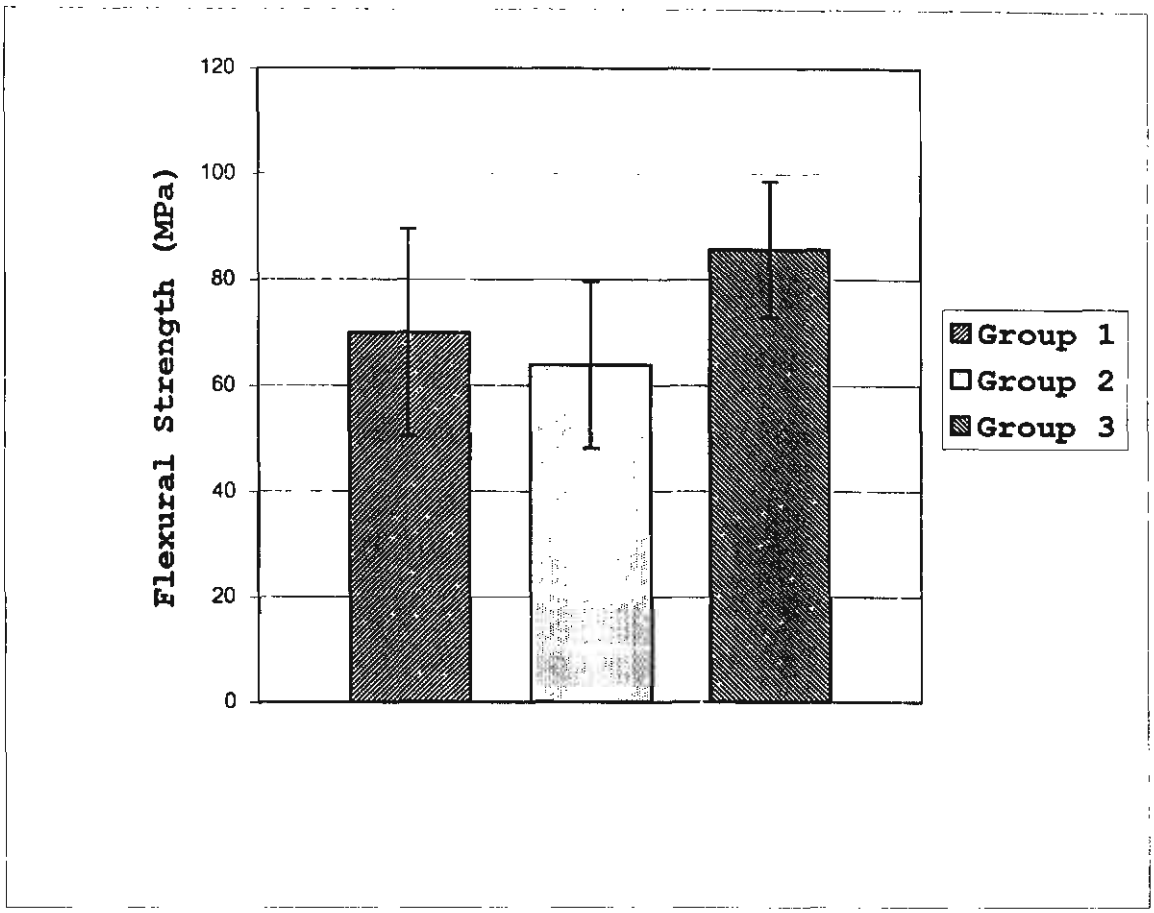

Figure 11: Bar graph representative of mean flexural strengths (Mpa) and standard deviation of IPS-Empress bars subjected to 3 different cooling conditions: Group 1) bars were allowed to a fast cooling in air; Group 2) bars were allowed to a fast cooling in water; and Group 3) bars were allowed to a slow cooling in air.

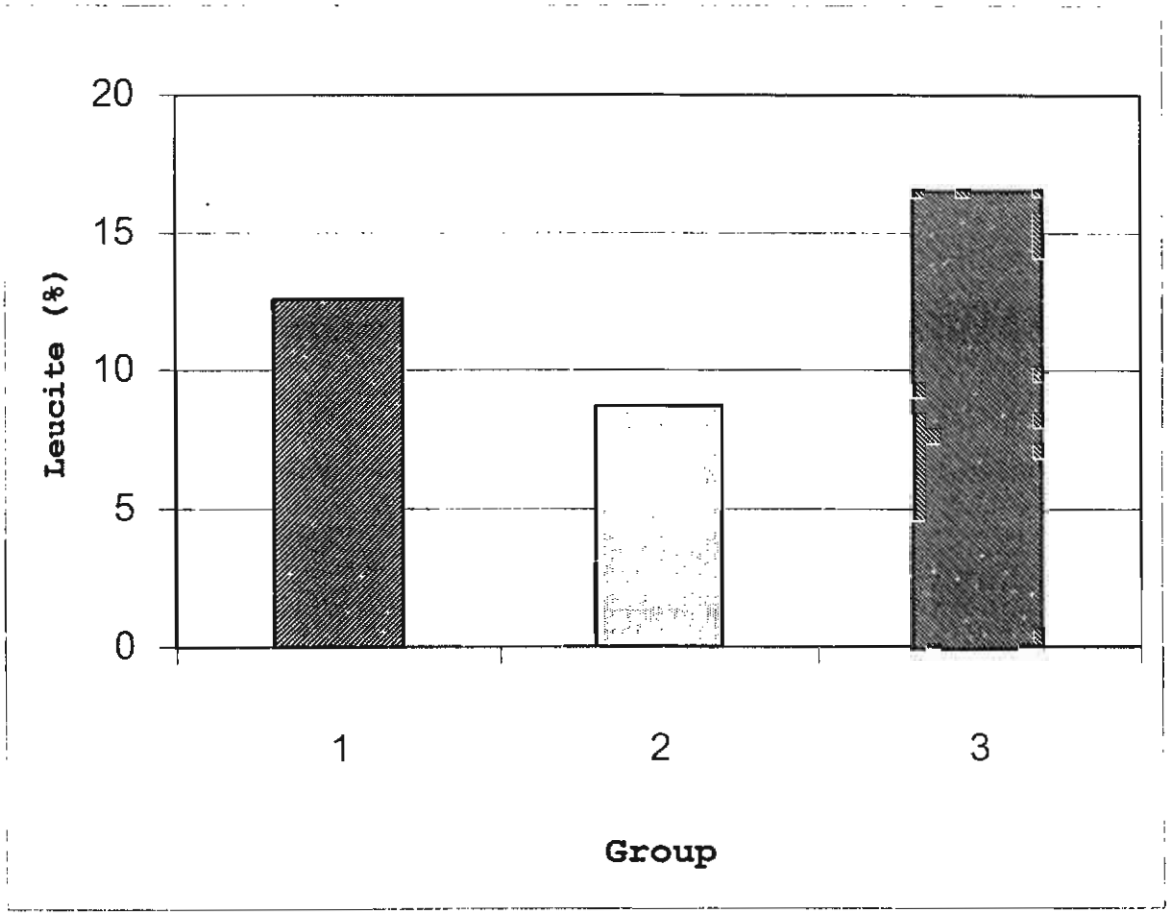

Figure 12: Bar graph representative of leucite content of IPS-Empress bars subjected to 3 different cooling conditions: Group 1) bars were allowed to a fast cooling in air; Group 2) bars were allowed to a fast cooling in water; and Group 3) bars were allowed to a slow cooling in air.

DISCUSSION

Leucite can be incorporated into many of dental porcelains from either the incongruent melting of feldspar or it can be added as a synthetic powder. Alteration of leucite content in dental porcelain may occur during multiple firing (Mackert, et al., 1996; Mackert, and Evan, 1991a), isothermal heat treatment (Mackert, and Evan, 1991a; Mackert, et al. 1995) and cooling (; Mackert, and Evan, 1991b).

The practice of slow cooling is carried out by some dental technicians in an effort to minimize the thermal shock encounter during withdrawal of the porcelain-fused-to-metal restoration from the hot furnace into a comparatively cool room air. The results of this study indicate that the increase in quantification of leucite crystals after a slow cooing is consistent with the increase in flexural strength. The higher the leucite content is thought to be responsible for increased flexural strength.

For all tested specimens, upon cooling, revealed only the low-temperature (tetragonal) form of leucite at room temperature, as shown in Figures 7-9. The low-temperature (tetragonal) and high-temperature (cubic)

forms coexisted at 400° C, having the volume fractions of tetragonal and cubic leucite of 0.78 and 0.22 respectively. Only cubic form was found at 600-700° C (Mackert et al., 1986).

A slow cooling rate has also been reported to produce additional leucite but the details of the formation and detection of the leucite was unclear (Mackert and Evans, 1991). Nevertheless, the increase in leucite content was found to increase the coefficient of thermal expansion of the glass matrix from 10.7 x 10^{-6}/°C to the 13-15 x 10^{-6}/°C.

In porcelain-fused-to-metal restorations, the high expanding leucite raises the bulk of veneering porcelain thermal expansion to a level where it is compatible with the metal substrate. Leucite crystals in all-ceramic restorations, in contrast to porcelain-fused-to-metal restorations, are not for thermal compatibility, but as a reinforcing material (Mackert and Russell, 1996).

Dong et al. showed that the injection molded system using a high leucite ceramic ingot produced better dispersion of the glassy matrix and attributed the improved strength comparing to the sintering process using to fabricate porcelain-fused-to-metal restorations.

As the porcelain cools down from the maximum firing temperature, the heated leucite crystals contract more

than the surrounding glassy matrix, owing to the their higher coefficient of thermal expansion and the cubic-to-tetragonal phase transformation. At the temperature below the glass transition temperature of the glass matrix, the walls of the cracks around the leucite crystals separate due to the contraction of the leucite particles. As a consequence, the strain of the contraction of the leucite crystals is not completely transmitted to the surrounding glass matrix, instead, this strain is relieved through the further opening of the cracks. The bulk thermal expansion of porcelain is thus influenced by the microcrack density of that porcelain, and any change in that microcrack density will be reflected in an altered bulk thermal expansion (Mackert et al., 1994). Therefore, the increase in flexural strength of the slow cooling group is associated with the increase amount of leucite crystals.

It has also been reported that the cooling conditions had little effect on the microstructural change of leucite crystals in the dental porcelain, since the leucite crystals were stabilized by the incorporation of additives into lattice (Ong et al., 2000; and Ban et al., 1998). Furthermore, the technique of heat pressing in an IPS-Empress system is not only a method of a shape forming it also has a positive influence on the strength

of the ceramic material. It is reasonable to suppose that the improved strength of pressed ceramic material resulted from the better dispersion of the leucite crystals in the glass matrix (Dong et al., 1992). Heat pressing, however, does not increase the leucite content of the IPS Empress ceramic.

The injection molded system is designed to fabricate all-ceramic restoration using either a surface staining (monolayer) or a layering (bilayer) technique. For a stainging technique, the heated ingot is injected into a mold of a full contour restoration. As a consequence, a monochromatic is performed. The surface coloration in combination with the shaded resin cement is suggested in order to match the color of the abutment tooth.

The layering technique consists of a fabrication of core and subsequently a veneering porcelain. Flexural strength and elastic modulus of a core material should be greater than those of a veneering porcelain in order to withstand the masticatory loading. It is because when the teeth of both arches are in contact, the maximum compression is at the occlusal surface (veneering surface), while the maximum tension is at the bonding surface (core material). Brittle materials, in general, would fail at the maximum tension. In addition, the fractographic analysis of this study showed the crack

origins of all tested bars were initiated at the tension side, as shown in Figure 10.

The result of this study suggests a slow cooling technique be appropriate for both surface staining and layering techniques. Even though a fast cooling in air is recommendded by the manufacturer, the quantification of leucite crystals in combination with the flexural strength is inferior comparing to a slow cooling technique.

CONCLUSIONS

1. The increase in quantification of leucite crystals after a slow cooing is consistent with the increase in flexural strength.

2. The higher the leucite content is thought to be responsible for increased flexural strength.

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APPENDIX

- - - - - O N E W A Y - - - - -

Variable STRENGTH flexural strength
By Variable GROUP empress

Analysis of Variance

Source	D.F.	Sum of Squares	Mean Squares	F Ratio	F Prob.
Between Groups	2	5101.1668	2550.5834	9.6324	.0002
Within Groups	57	15093.1911	264.7928		
Total	59	20194.3579			

Group	Count	Mean	Standard Deviation	Standard Error	95 Pct Conf Int for Mean
Grp 1	20	69.9667	19.5159	4.3639	60.8330 TO 79.1005
Grp 2	20	63.8804	15.7723	3.5268	56.4987 TO 71.2620
Grp 3	20	85.7598	12.8352	2.8700	79.7527 TO 91.7669
Total	60	73.2023	18.5007	2.3884	68.4231 TO 77.9815

GROUP	MINIMUM	MAXIMUM
Grp 1	33.7520	98.6910
Grp 2	37.8770	96.1080
Grp 3	64.6300	105.1650
TOTAL	33.7520	105.1650

Levene Test for Homogeneity of Variances

Statistic	df1	df2	2-tail Sig.
2.3108	2	57	.108

- - - - - O N E W A Y - - - - -

```
     Variable  STRENGTH   flexural strength
  By Variable  GROUP      empress

Multiple Range Tests:  Tukey-HSD test with significance le/el .050

The difference between two means is significant if
  MEAN(J)-MEAN(I)  >= 11.5064 * RANGE * SQRT(1/N(I) + 1/N(J))
  with the following value(s) for RANGE: 3.40

   (*) Indicates significant differences which are shown in the lower triangle

                          G G G
                          r r r
                          p p p

                          2 1 3
    Mean      GROUP

   63.8804    Grp 2
   69.9667    Grp 1
   85.7598    Grp 3       * *

 Homogeneous Subsets (highest and lowest means are not significantly different)

Subset 1

Group       Grp 2         Grp 1

Mean       63.8804       69.966
- - - - - - - - - - - - - - - - - -

Subset 2

Group       Grp 3

Mean       85.7598
- - - - - - - - - -
```

		[½2Õ]	[Counts]	[s]	[½2Õ]	[Counts]	[s]
uecite	PEAK	27.21	22279.00	50.0	26.31	4778.00	50.0
					28.31	4504.00	

Phase name	Concentration [%]	Net intensity [counts/s]
luecite	16.621	401.104

Results: Sample A

MRD. PC-APD, Diffraction software

PI file name: DENT
Quantify program: DENT
Model: STRAIGHT LINE

Phase name	Concentration [%]	Net intensity [counts/s]
luecite	16.621	401.104

ntensity list: Sample B

MMRD. PC-APD, Diffraction software

PI file name: DENT
Diffractometer: PW3710 based
X-ray tube: Cu LFF (Long Fine Focus) 40 kV 30 mA
Divergence slit: 1½
Spinner: OFF
Quantify program: DENT
Model: STRAIGHT LINE

Phase ame	Phase type	Phase angle [½2Õ]	Phase intens. [Counts]	Meas. time [s]	Backgr. angle [½2Õ]	Backgr. intens. [Counts]	Backgr. time [s]
luecite	PEAK	27.21	13677.00	50.0	26.31	5035.00	50.0
					28.31	3726.00	

Phase name	Concentration [%]	Net intensity [counts/s]
luecite	8.705	210.086

ntensity list: Sample B

MRD. PC-APD, Diffraction software

PI file name: DENT
Diffractometer: PW3710 based
X-ray tube: Cu LFF (Long Fine Focus) 40 kV 30 mA
Divergence slit: 1½
Spinner: OFF
Quantify program: DENT
Model: STRAIGHT LINE

hase ame	Phase type	Phase angle [½2Õ]	Phase intens. [Counts]	Meas. time [s]	Backgr. angle [½2Õ]	Backgr. intens. [Counts]	Backgr. time [s]
luecite	PEAK	27.21	13795.00	50.0	26.31	5093.00	50.0
					28.31	4623.00	

Phase name	Concentration [%]	Net intensity [counts/s]
luecite	8.406	202.859

ntensity list: Sample A

MRD. PC-APD, Diffraction software

PI file name: DENT
Diffractometer: PW3710 based
X-ray tube: Cu LFF (Long Fine Focus) 40 kV 30 mA
Divergence slit: 1½
Spinner: OFF
Quantify program: DENT
Model: STRAIGHT LINE

ase ame	Phase type	Phase angle	Phase intens.	Meas. time	Backgr. angle	Backgr. intens.	Backgr. time

X-ray tube: Cu LFF (Long Fine Focus) 40 kV 30 :
Divergence slit: 1½
Spinner: OFF
Quantify program: DENT
Model: STRAIGHT LINE

Phase name	Phase type	Phase angle [½2Õ]	Phase intens. [Counts]	Meas. time [s]	Backgr. angle [½2Õ]	Backgr. intens. [Counts]	Backgr. time [s]
luecite	PEAK	27.33	102339.00	50.0	26.31	5037.00	50.0
					28.31	5631.00	

Phase name	Concentration [%]	Net intensity [counts/s]
luecite	103.011	2207.526

Intensity list: standard 100%luecite

MMRD. PC-APD, Diffraction soft

PI file name: DENT
Diffractometer: PW3710 based
X-ray tube: Cu LFF (Long Fine Focus) 40 kV 30 mA
Divergence slit: 1½
Spinner: OFF
Quantify program: DENT
Model: STRAIGHT LINE

Phase name	Phase type	Phase angle [½2Õ]	Phase intens. [Counts]	Meas. time [s]	Backgr. angle [½2Õ]	Backgr. intens. [Counts]	Backgr. time [s]
luecite	PEAK	27.28	96582.00	50.0	26.31	4159.00	50.0
					28.31	5318.00	

Phase name	Concentration [%]	Net intensity [counts/s]
luecite	87.422	2090.623

Intensity list: Sample C

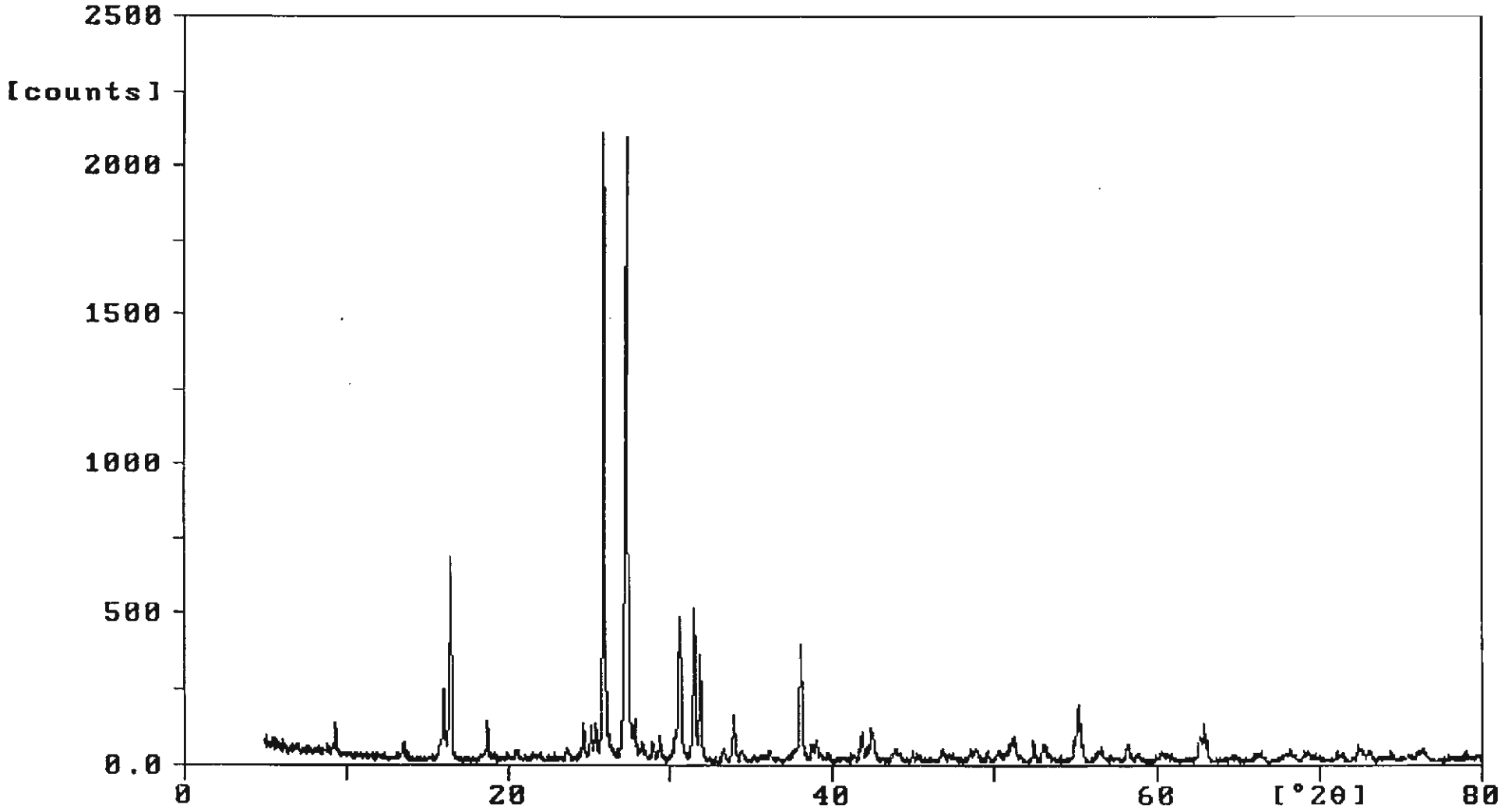
MMRD. PC-APD, Diffraction softwa

PI file name: DENT
Diffractometer: PW3710 based
X-ray tube: Cu LFF (Long Fine Focus) 40 kV 30 mA
Divergence slit: 1½
Spinner: OFF
Quantify program: DENT
Model: STRAIGHT LINE

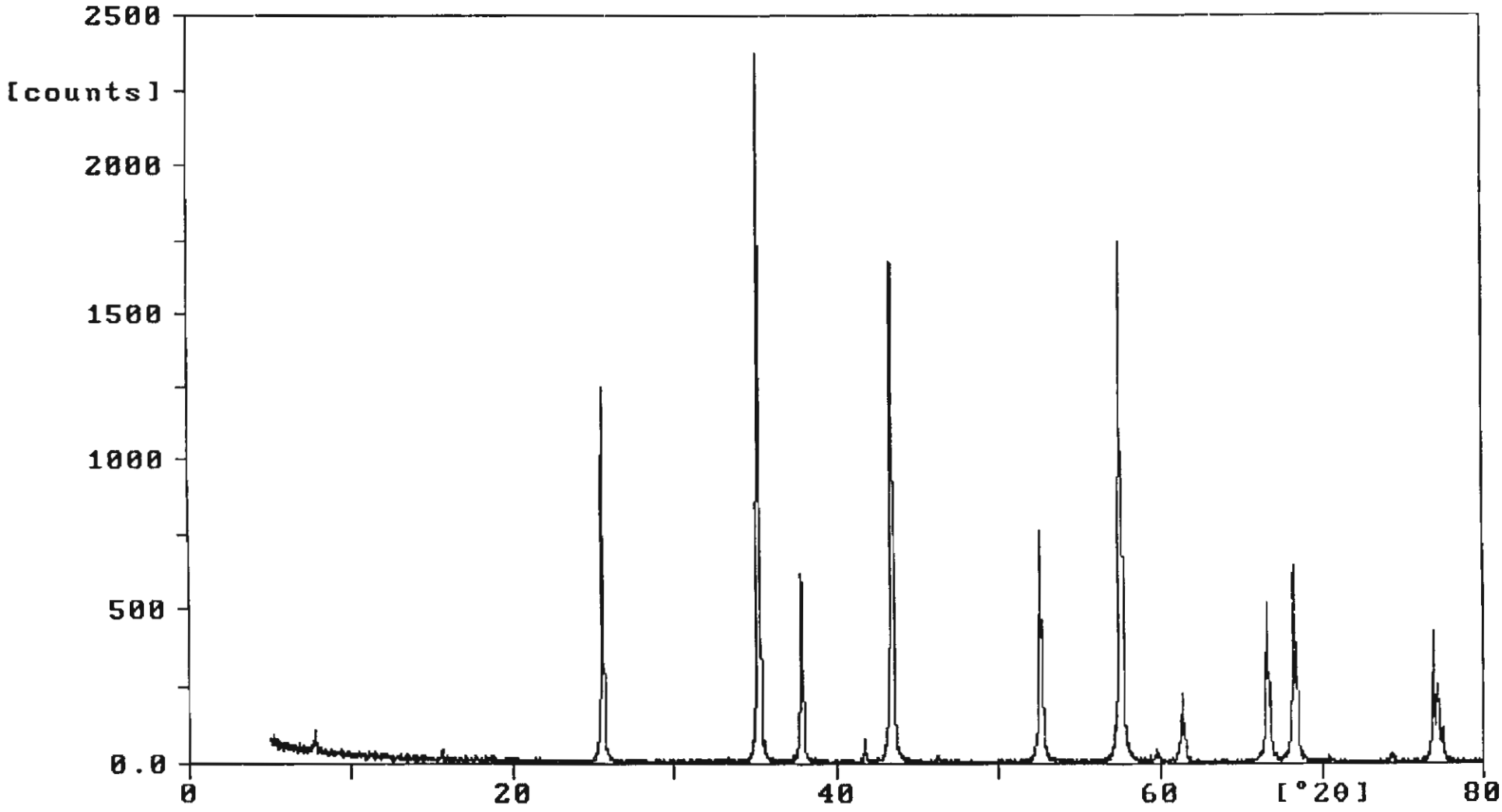
Phase name	Phase type	Phase angle [½2Õ]	Phase intens. [Counts]	Meas. time [s]	Backgr. angle [½2Õ]	Backgr. intens. [Counts]	Backgr. time [s]
luecite	PEAK	27.21	19114.00	50.0	26.31	6486.00	50.0
					28.31	4859.00	

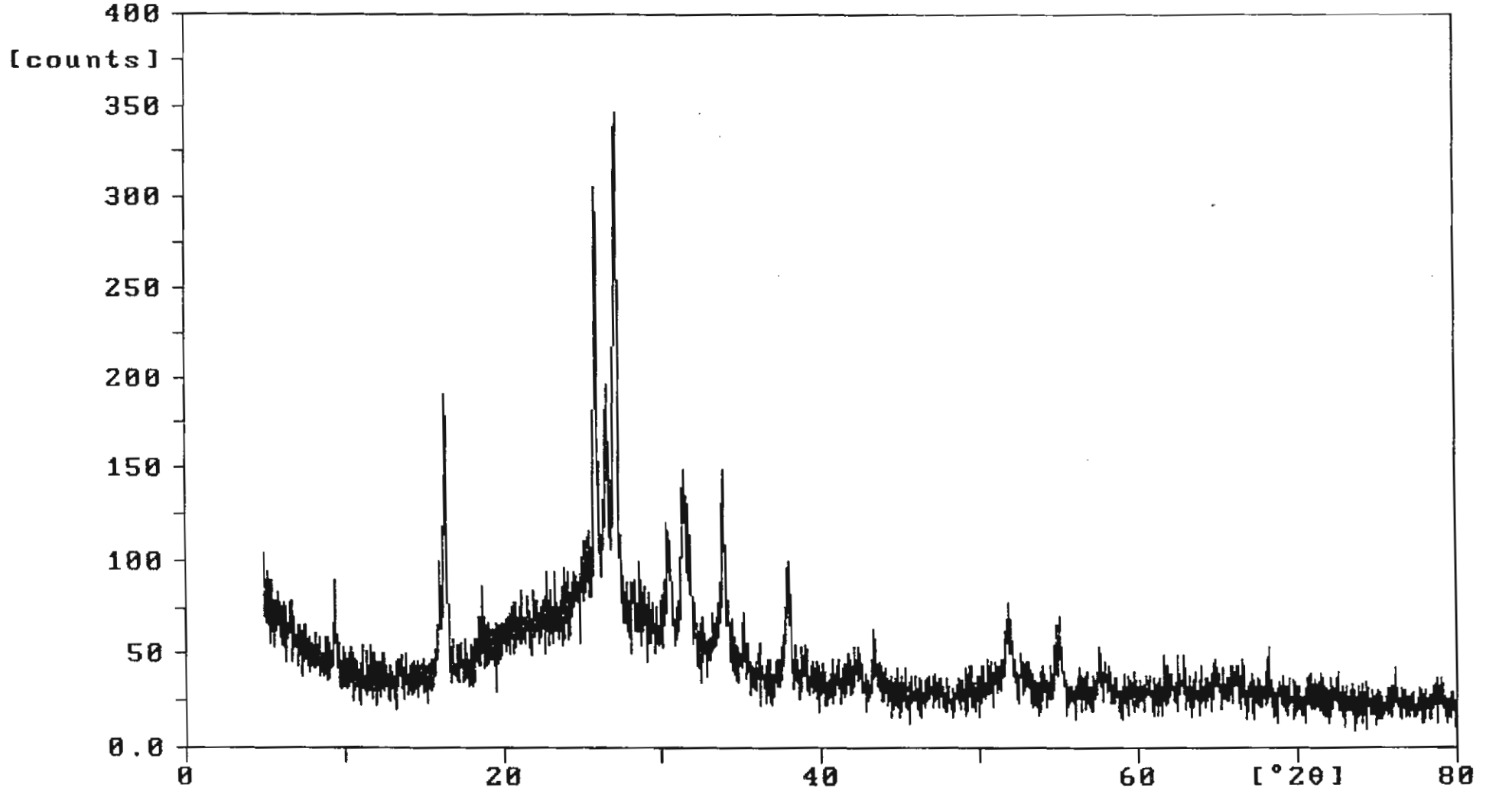
Phase name	Concentration [%]	Net intensity [counts/s]
luecite	12.599	304.058

Sample identification: luecite



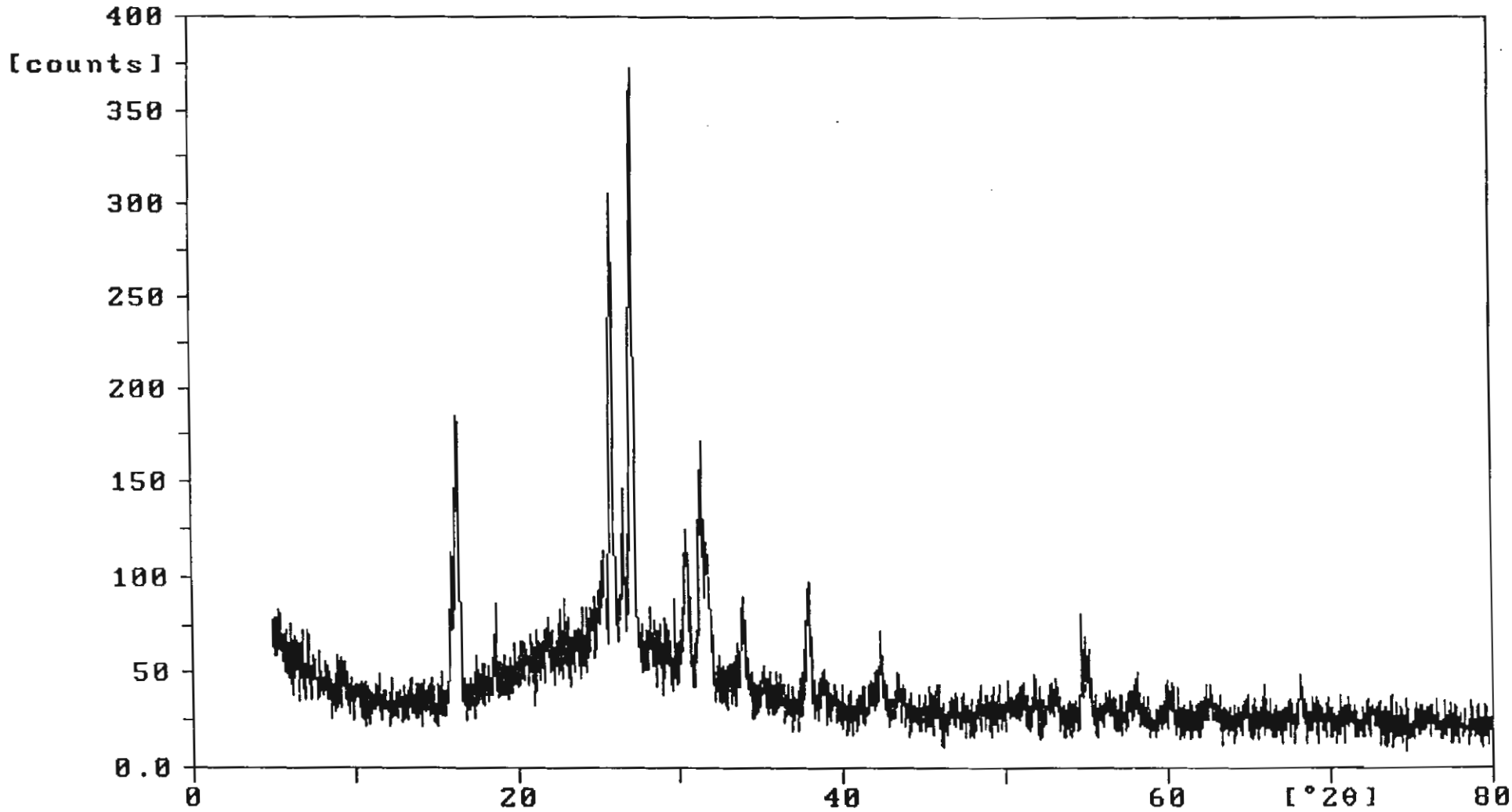
Sample identification: corundum





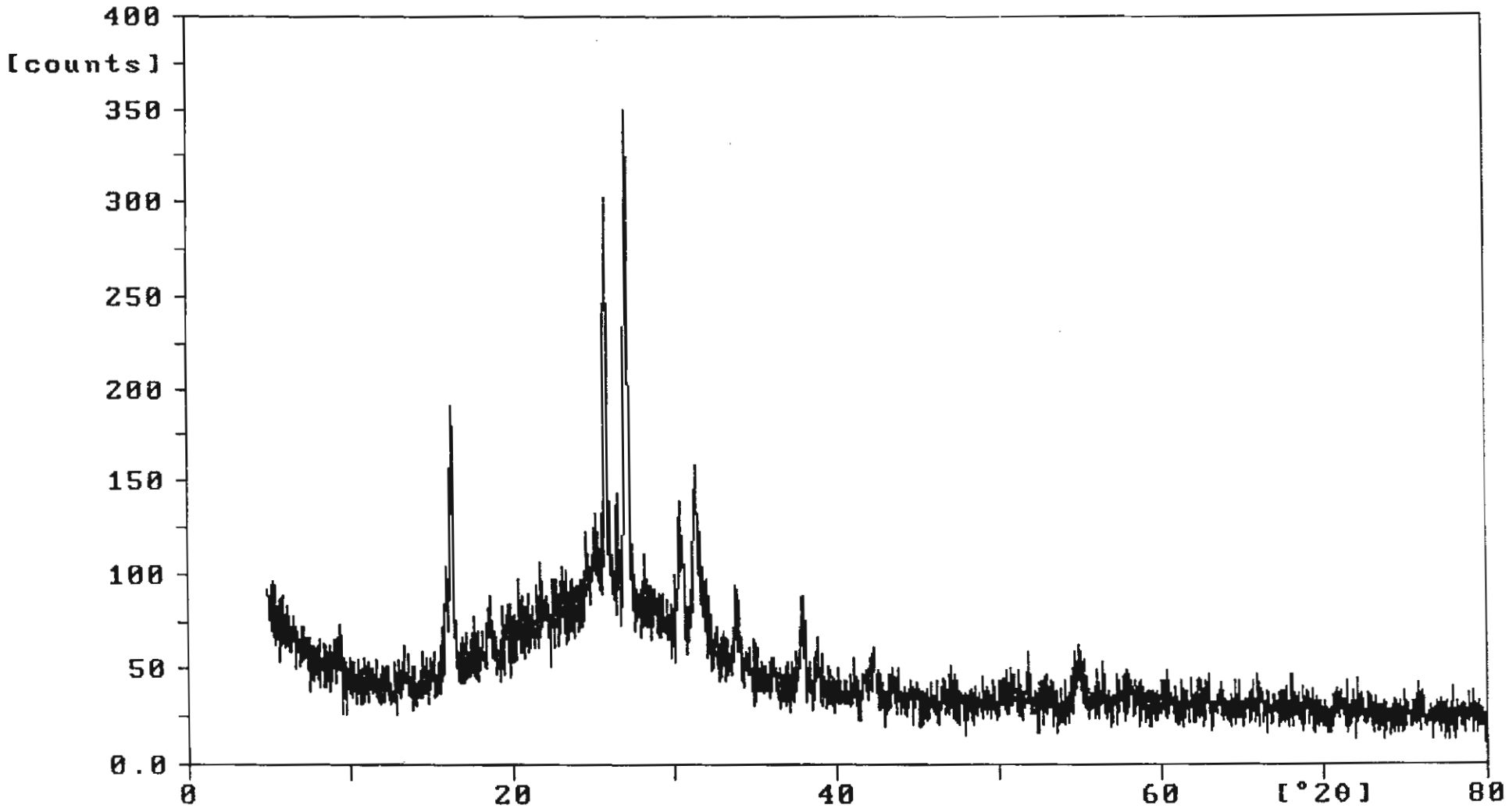
IPS-Empress bars were subjected to a fast cooling in air.

Sample identification:



IPS-Empress bars were subjected to a fast cooling in water.

Sample identification:



IPS-Empress bars were subjected to a slow cooling condition.

Intensity (a.u)
Leucite 12.6%
Leucite 8.7%
Leucite 16.6%
Cu - Kα 2 θ (degree)
20
40
60
80