

THE PORTLAND CEMENT AGGREGATE BOND: INFLUENCE  
OF SURFACE AREA OF THE COARSE AGGREGATE AS A FUNCTION OF LITHOLOGY 213

by

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(The opinions, findings, and conclusions expressed in this report are those of the author and not necessarily those of the sponsoring agencies.)

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## SUMMARY

A direct tensile test for measuring the bond of rock or mineral surfaces to portland cement paste, or for measuring the tensile strength of neat paste or of mortar specimens, has been devised using commercially available gripping devices and preparation equipment. The method of specimen preparation and testing permits the sample to be totally immersed in water throughout its entire history from casting to failure in the testing machine.

The results of a series of bond tests on selected materials indicate the following relative order of bonding strength to cement paste: frosted glass, quartz, limestone, neat cement paste, dolomitic marble, augite.

Scanning electron micrographs representative of the minerals used in the bond tests are presented with the conclusion that the apparently smooth surfaces of the minerals have high roughness factors and possess many times the apparent surface area. The importance of roughness factor in influencing the strength of the cement aggregate bond is analyzed, and the differential capacity (an electrochemical) method for determining roughness factors is identified as the most promising technique available.



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INTRODUCTION

The strength of concrete depends primarily on the strength of the cement paste matrix, the strength of the individual aggregate particles, and the strength of the bond between them — at the cement aggregate interface. Good evidence has been offered that the paste, or mortar, to aggregate bond is the weakest of these three potential failure links (Hsu, et al. 1963). And, in the course of investigations aimed at illuminating the sources of strength and durability in concrete, it was discovered that, all other things being equal, the strength of the cement aggregate bond varied with the type of rock being used as aggregate (Alexander 1959; Valenta 1961; Hsu and Slate 1963).

Significant and consistent differences in bond strength were found by Valenta between rocks in a group which included basalt, granite quartzite, dolomite, greywacke, and limestone. Within this group two basalts formed the strongest bonds, quartzite and dolomite intermediate bonds, and limestone, greywacke, and granite the lowest strength bonds, on fractured surfaces and using an indirect tensile test. Alexander also found definite differences in bond strength between various lithologies, but using only two as an example, found at 28 days a difference of 974 psi in bond strength between a chert (1777 psi) and a rhyolite (803 psi) based on 20 samples. That a real difference among the rock types exists is further substantiated by Alexander's finding that "no evidence was found of significant cement-aggregate bond strength differences between different samples of the same rock type." Furthermore, it was found that different rocks with apparently similar surface textures manifested very different cement aggregate bond strengths. Thus it appears that bond strength development is dependent upon some feature of the rock which is not readily apparent.

These previously published researches (Alexander, Valenta, and others) were concerned only incidentally with the actual interface between coarse aggregate and mortar; i. e., they were concerned only insofar as the interface influenced some other characteristic which interested them. For example, Valenta was interested in the significance of the bond for the durability of concrete and Alexander in the

explanation of the process of tensile failure of plain concrete. Two studies by Hsu, et al. (1963), one dealing with tensile bond strength and the other with microcracking and the stress-strain curve, indicate that of the three previously mentioned links in the chain of strength of concrete the bond between the paste and the aggregate is the weakest. Furthermore, Hsu, et al. (1963), examining thin slices of concrete by newly developed techniques, found that "cracks at the interface between coarse aggregate and mortar are widespread even in nonloaded concrete."

In view of these findings the investigation of the interface acquires a double significance — the first concerning the effect of different lithologies, and the second concerning the most vulnerable link in the chain of strength of concrete.

In spite of these results suggesting an interesting new problem, the solution of which might have a very practical application, previous workers were disinclined to investigate the bond from a materials point of view; that is, they were not so interested in the cause of the difference in bond as in the effect of the difference on certain other properties of concrete.

#### PURPOSE AND SCOPE

It is now apparent that the basis for a study such as is reported here existed and, accordingly, it was the intent of this research to investigate the reasons for the differences in bond strength between portland cement paste and different rocks, and to relate the differences to some property of the rock surface that is in turn a function of the differences in mineralogical and textural composition.

It is not contended that the nature of the cement aggregate bond has been investigated; that is, the study has not been concerned with the fundamental reason for the adhesion between rock surfaces and portland cement paste. Rather, it has been concerned with the reason for the differences in the degree of adhesion to different types of rocks. However, it is recognized that the data developed may support, or detract from, or encourage speculation regarding existing or new ideas on the mechanism and nature of the bond.

#### APPROACH AND PROCEDURES

In developing an approach to the problem it was necessary to hypothesize that it is indeed some property of the surface of a fractured or cut rock that influences the strength of the bond between the aggregate and the paste. Assuming this

hypothesis, a simple analysis of the strength of an adhesive joint between any two substances indicates that the rupture strength of the joint is the product of the (unit) bond stress of the adhesive, and the surface area over which the adhesive acts.

The unit bond stress, loosely speaking, is a measure of the stickiness of the adhesive substance. Some adhesives are stickier than others and thus, qualitatively speaking, a very adhesive substance acting over a small area will support the same weight as a less adhesive substance acting over a larger area. There are, therefore, two contributing elements to the strength of a bond. The former, the bond stress, has to do with the fundamental adhesive nature of the substance and, as previously mentioned, this element was investigated in this study. The latter property — the amount of surface area over which the adhesive — in this case portland cement paste — acts, formed the basis for the approach to the problem.

If different rocks, on any kind of surface whether it be sawn, polished, or fractured, possess different true surface areas per unit of projected or cross sectional area — that is, different roughness factors\* — then they will, after contacting an adhesive, exhibit different rupture strengths at the interface. It is hypothesized that it may be this effect which is responsible for the observed differences in the strengths of the bond of different rock types to cement paste.

Rough surfaces are sometimes assumed to have more surface area than smooth surfaces, but this is not necessarily true as the following simple diagram illustrates.




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\* The roughness factor is the true available surface area divided by the geometric or projected area.

In the diagram AB is a smooth line segment and CD is rough. CD with 45° saw tooth projections has approximately  $1\frac{1}{2}$  times (or 50%) more line in the same distance\*. Now if the 45° projections on CD were microscopic or submicroscopic, that is to say, if there were a great number of them and the surface appeared as smooth as AB, the geometrical result would be exactly the same; i. e. there would be  $1\frac{1}{2}$  times more line between C and D than between A and B. Thus there would be the same surface area but essentially no roughness.

In view of these observations it is evident that any measurement of, or related to, simple roughness will not suffice to represent the true surface area.

The research approach, therefore, was to correlate bond strength determinations on selected rocks and minerals with roughness factor determinations on the same materials. Additionally, in order to provide insight into the question of how the roughness factor relates to detailed surface morphology, scanning electron micrographs of the surfaces of the materials used in the study were compared.

The investigation therefore was divided into three parallel sub-topics:

- (1) The development of a method for determining the bond strength of portland cement paste to rock or mineral surfaces; and the determination, by that method, of the bond strength of selected rocks and minerals.
- (2) The identification, or development, of a method for the determination of roughness factors of rock and mineral surfaces; and the determination, by that method, of the roughness factors of those minerals and rocks used in the bond strength tests.
- (3) The examination of the surface micromorphology of the specimens used in the test program by means of the scanning electron microscope.

The main point in the analysis of the data presented later will be to compare the relative rankings of the tensile bond strengths to cement paste of the test materials with their relative rankings with respect to the roughness factor. If those materials with high tensile bond strength also have high roughness factors and correspondingly those with low strengths have low roughness factors and no other high-low combinations complicating the situation are present, then the evidence will be good that the roughness factor is a strong determinant of bond strength. If, however, the two other qualitative combinations are present, viz., low strength with a high roughness factor and high strength with a low roughness factor, then the indication will be that other variables, e. g. the surface chemistry of the mineral, are also strong determinants of bond strength.

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\* The increase is strictly  $\sqrt{2}$  times the base distance.



### Development of Bond Test Method

Aggregate-cement bond strength has been determined by many different procedures to suit the requirements of particular investigations and the availability of equipment (Alexander et al. 1965). One of the objectives of this portion of the study was to develop a test procedure which would meet both the scientific and engineering requirements for a proper bond test and at the same time be economical of materials, equipment, and time.

The desirability of testing the strength of an adhesive bond by a direct tensile method, if possible, is well-known and need not be discussed at length here. Briefly, it is only by direct or uniaxial tension that the component of strength due to adhesion can be isolated and evaluated free of effects contributed by mechanical interlocking, friction, and so forth.

The test developed in this investigation combines modifications of the methods used by Scholer (1965) for specimen preparation and by Dale and Ludwig (1965) for direct tensile testing.

#### Specimen Preparation

An approximately 5/16-inch slice of mineral or rock is cut with a diamond slabbing saw and trimmed to approximately 4 x 4½ inches. It may be irregular in shape provided it does not exceed 4 inches across. The slice is ground on both sides with a medium abrasive (75 micron) to eliminate weakly adhering flakes and shattered crystal debris. It is then stored under water until the time of casting of the specimen block.

In order to prepare the specimen block from which the cylinders to be tested in tension will be drilled, the following procedure is used.

Braces are prepared to fit across the top of a standard beam mold between which the specimen slice can be grasped so that it will hang down into the mold perpendicular to the sides and parallel to the ends. End plates are used to divide the mold into portions such that two 4-inch cubes can be cast at one time, and the braces are located such that the specimen slice will depend into the middle of a 4-inch block with clearance on the bottom and sides and a ½-inch or so "tab" protruding between and above the braces. The braces must be adjusted so that perpendicularity of the specimen slice is assured; it is checked during the setting up procedure with a machinist's combination square (Figure 1). When the slice is in position a .35 water to cement (Type II) ratio paste is cast vertically on both

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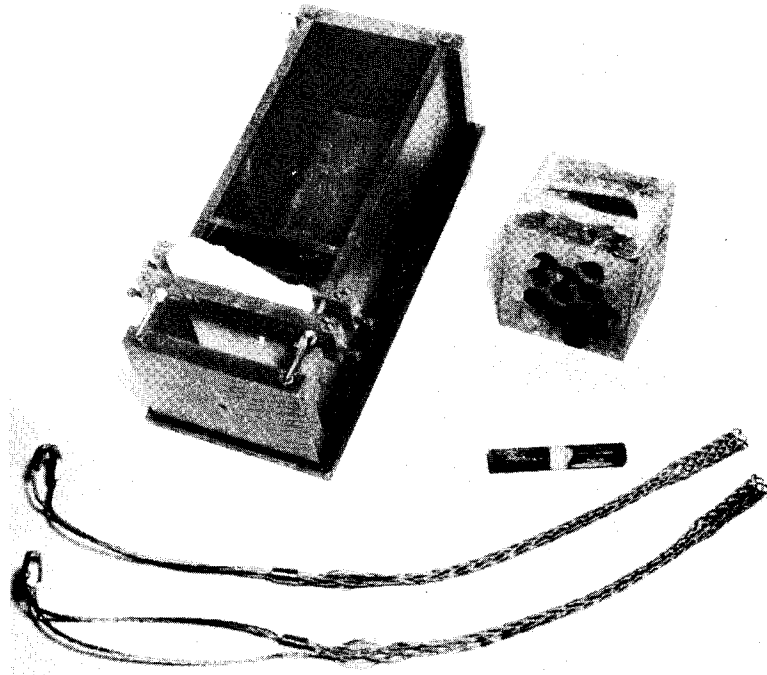


Figure 1. Standard beam mold with braces in place holding mineral slice (left). Unmolded specimen block which has been partially cored (right). Wire mesh grips and cylindrical bond test specimen in foreground.

sides of the hanging slice and the mold is vibrated\*. The mold is stored in a moist room for 24 hours and then unmolded. The specimen is now a sandwich with a slice of mineral or rock between approximately 1-7/8 inches of paste on each side; the former top edge of the mineral slice protrudes above the paste. The specimen block is now stored under lime saturated water for about 25-27 days until the next step in the preparation.

When the specimen has reached adequate strength it is removed from the lime water and approximately twenty  $\frac{1}{2}$ -inch diameter cylinders are drilled, in water, perpendicular to the mineral slice, and stored under lime water until the time of testing. The core drill and associated equipment are essentially the same as used by Scholer (1964 and 1965). At this point the specimen differs from that used by Scholer in that it is a cylindrical sandwich of mineral, or rock, and cement paste, rather than a cylinder which is half paste and half rock.

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\* Note, the same barrel of Type II cement was used throughout the study.

On the 28th day (in the procedure followed in the present study) the cylindrical specimens are tested by a direct tensile method described by Dale and Ludwig (1965), in which the paste ends of the specimen cylinder are mounted in wire mesh grips (Ingersoll holders) and pulled in a testing machine. An important addition to the method, in recognition of the desirability of testing the specimen in as uniformly moist a condition as possible, was the use of a plexiglass container with a through-connection at the bottom to receive the end of the wire mesh grip. The container is filled to a suitable level with water so that the specimen is completely immersed during the tensile testing procedure. Figure 2 shows a specimen in the wire mesh grips immersed in water while the load is being applied. The rate of loading for all specimens was approximately 750 psi/minute.

Figure 3 shows the arrangement in place in the testing machine.

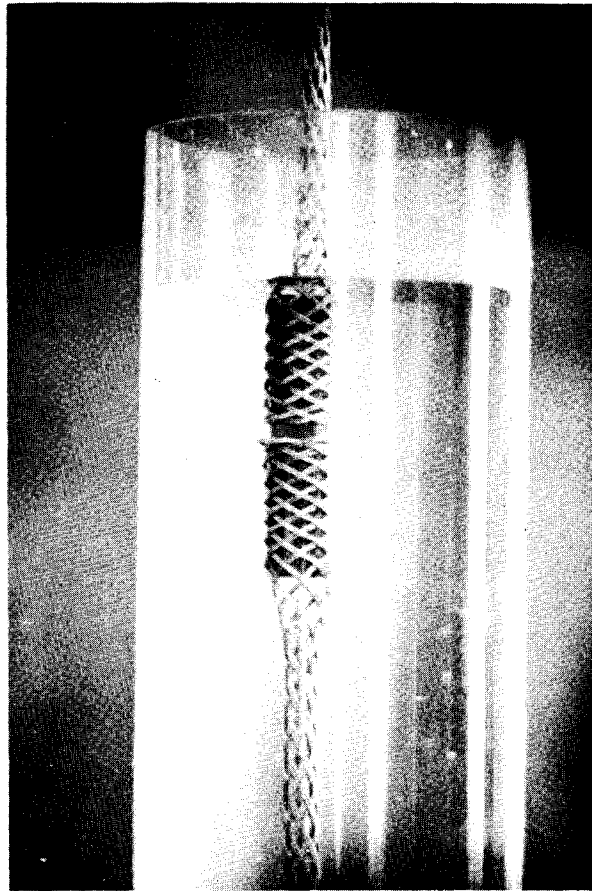


Figure 2. Bond test specimen under tension.

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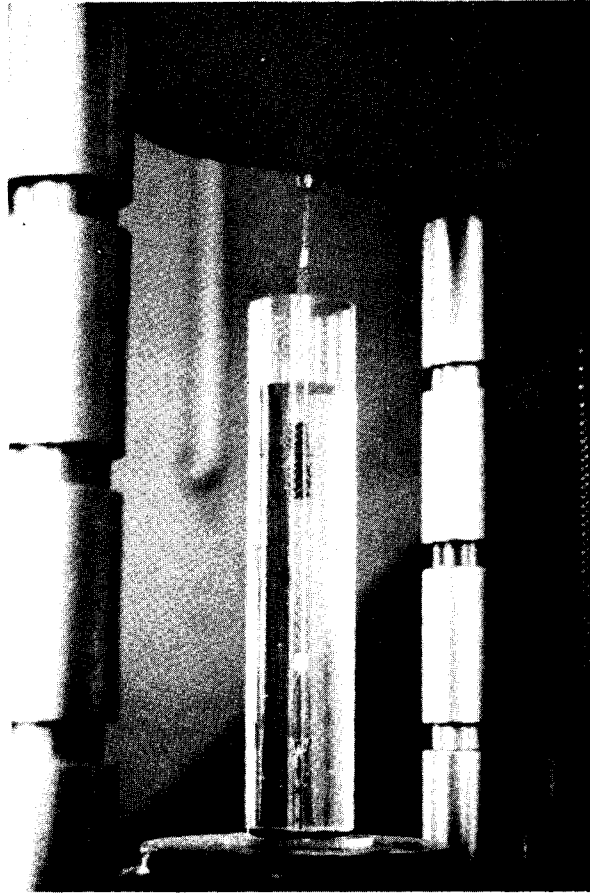


Figure 3. Bond test specimen in wire mesh grips under water in plexiglass container in testing machine.

#### Minerals and Rocks Used in the Investigation

In the first phase of the testing program the intention was to use a selection of relatively pure minerals which would represent volumetrically some of the most important common rock forming minerals. These would include quartz, feldspar, olivine, pyroxenex (augite), amphibole (hornblende), calcite, and dolomite. Micas, although important rock forming minerals, were omitted from consideration because of the anticipated difficulty in processing and preparing suitable samples perpendicular to the plane of cleavage. Minerals were chosen in preference to mixtures of minerals, that is rocks, in order to avoid considerations related to isolating and assessing the contribution of individual mineral species to the total surface area. However, in preparatory, or pilot, studies to develop the method of sample fabrication a pure fine-grained limestone was used. After a very short initial experimental series the whole procedure worked so well that the limestone results were included in the data.

Additionally, out of scientific curiosity and for purposes of comparisons with natural materials, a hardened neat cement paste slice and a frosted pane of ordinary window glass were used in bond test specimens.

Specimen blocks of olivine and microcline did not, during coring, yield enough cylinders for a proper test.

The results of the tensile tests are summarized in Table I.

TABLE I

SUMMARY OF RESULTS OF PASTE-MINERAL (OR ROCK) BOND TESTS

Type II Cement, W/C = .35<sup>a</sup>, rough ground surface, 28 days<sup>a</sup>

Sample	$\bar{X}$	V	nt	Notes
Frosted Glass	627	32	12	
Quartz	548	29	19	
Cement Paste <sup>b</sup>	494	40	21	Tested at 14 days
Cement Paste <sup>b</sup>	493	33	22	
Limestone	503	34	25	Combined cores from 2 blocks
Limestone	450	27	17	W/C = .40
Limestone	437	40	26	Combined cores from 2 blocks
Dolomitic Marble	454	28	15	The dolomitic marble specimens are three companion blocks of exactly the same materials.
Dolomitic Marble	267	13	11	
Dolomitic Marble	267	12	11	
Augite	323	29	10	

$\bar{X}$  — mean value in pounds per square inch to cause bond failure

V — coefficient of variation for the cores successfully tested, in percent

nt — number of cores

<sup>a</sup> Except as noted

<sup>b</sup> Slices of cement paste from a broken block were used as "aggregate".

### Discussion of Test Results

The tensile test results indicate the following relative rankings for strength of bond to cement paste:

- (1) frosted glass,
- (2) quartz,
- (3) hardened neat cement paste (w/c = .35)
- (4) limestone,
- (5) dolomitic marble, and
- (6) augite.

It is apparent that the more siliceous materials adhered better to portland cement paste than the less siliceous. This observation may lend support to Alexander's (1964) conclusion that the bond between siliceous aggregate and portland cement is pozzolanic in origin; that is, it derives from a reaction of silica with free lime or calcium hydroxide in the presence of water. Alternatively, it may be the case that the more siliceous materials do, in fact, have greater roughness factors. However, there still must be a process of adhesion, which is presumably not pozzolanic in nature, and which will account for the bond between, for example, carbonate minerals and portland cement paste.

### Identification of the Method for Surface Area and Roughness Factor Determinations

The roughness factor is the ratio of the B. E. T. surface area to the projected geometric area. Therefore it is a measure of the total area available for reaction or adsorption (or coating, as by an adhesive) per unit of simple cross sectional area.

For purposes of this investigation, however, the determination of the B. E. T. surface area in the manner in which it is conventionally determined and expressed, that is, in square meters per gram of material, is not useful. The reasons are that in this study the simple dimensions of the surface whose B. E. T. area is being determined must be accurately known and the contribution of any other surfaces available for gas adsorption, e. g., pores and cracks, must be avoided. Therefore a powdered specimen, which might typically be used for an industrial type determination of specific surface, is not suitable. Also massive samples, as is done with hydrated compacts of portland cement, for which the total surface area, both internal and external, is determined, are not suitable.

In summary, in order to determine the roughness factor one must be able to isolate a particular surface of accurately known dimensions and determine the total area on that surface only.

Certain techniques permitting a straightforward determination of the roughness factor have been developed and are being used by electrochemists for determining the true surface area of solid electrodes (Brodd and Hackerman 1957). The basis of the technique is that under certain specific conditions the differential capacitance of, for example, a pure platinum electrode, is approximately 20 microfarads  $\text{cm}^2$ . Therefore, by measuring both the capacitance and the dimensions of the surface, the roughness factor may be calculated directly. This approach as applied to the problem of determining the roughness factors of minerals requires simply that the mineral surface be coated with platinum, in a fashion essentially the same as for scanning electron microscopy, and the capacity of the surface measured in an apparatus essentially similar to that which has been described in the literature (Brodd and Hackerman 1957; Stoner and Srinivasan 1970; Gileadi, Tshermikovskiy, and Amdursky 1968).

In practice, the particular electrodes and plating material used must be calibrated, the surface not to be included in the measurement must be masked or insulated, and an electrical contact must be maintained between the platinum coated surface and the electrolyte solution in which the sample is immersed. The objectives of this investigation do not require, in this instance, absolute values for surface area or roughness factor determinations. Relative values, so that the specimens may be ranked according to roughness factors, will provide data for the comparison with the relative tensile bond strengths.

#### Scanning Electron Microscopy

The samples prepared for scanning electron microscopy were small cylinders (5-mm diameter) of quartz, dolomite, microcline, albite, augite, and olivine drilled from slabs used for preparing the bond test specimen blocks. The cylinders were broken across to a length of approximately 5 mm and the naturally fractured surfaces, after coating with gold-palladium, were examined with the scanning electron microscope. Three specimens each were mounted on specimen stubs — this arrangement facilitated rapid comparison between samples. Also, prior to the SEM examination a full face photograph of each specimen cylinder was taken and enlarged to an 8 x 10 print. The purpose of this photograph is to serve as a "map" for delineating areas on the specimen from which high magnification photographs will be taken. By this technique, the microtopography of very small areas may be related to domains of differing morphology as seen under low magnification and an estimation may be made in this way of the relative proportions of areas of differing micromorphology. Selected views representing the most frequently observed types of surfaces for each mineral species are presented in Figures 4 through 16.

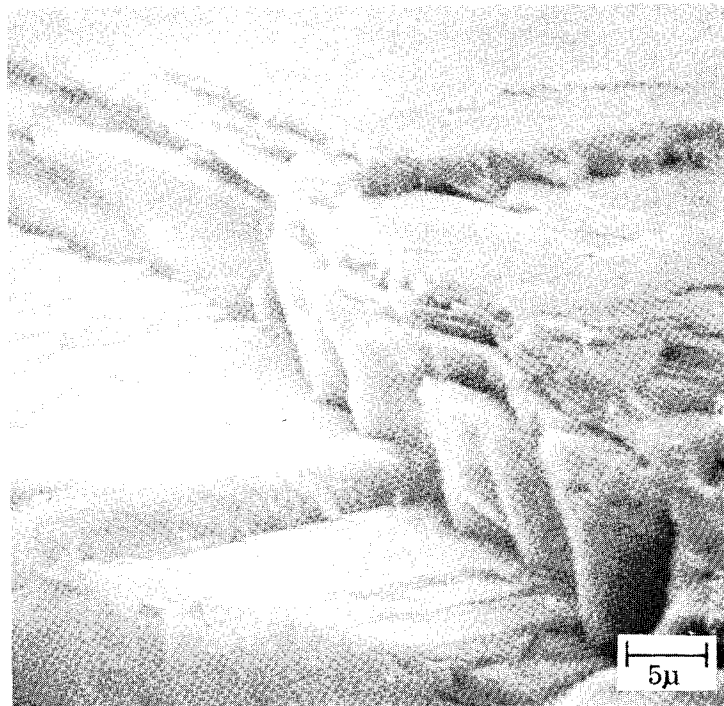


Figure 4. Fractured surface of dolomite. Magnification 2k, showing region of typical rhombohedral cleavage.

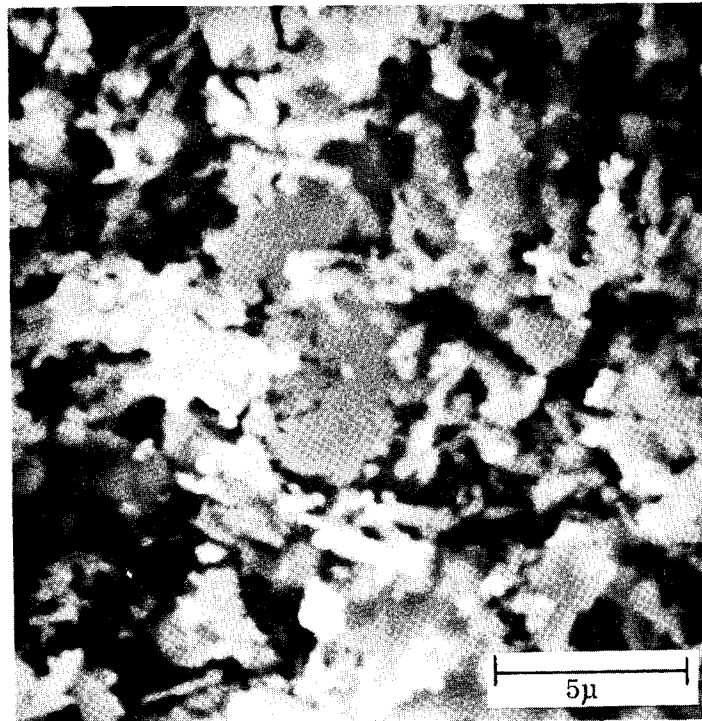


Figure 5. Same specimen as Figure 4 showing a region of intricate and rough morphology, which is typical of much of the surface. Magnification 5k.



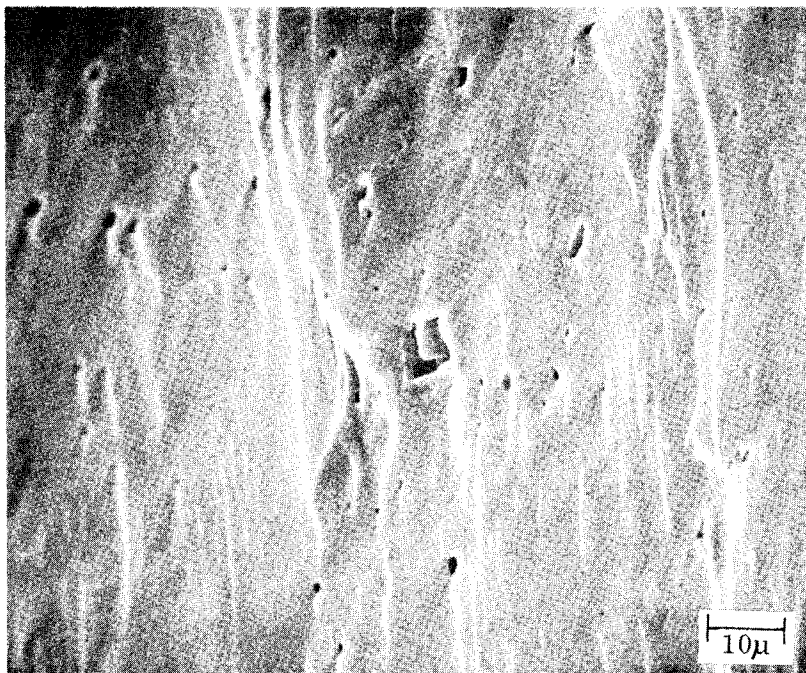


Figure 6. Surface of quartz, 100 x, typical view of porous low relief surface showing apparently smooth planar areas.

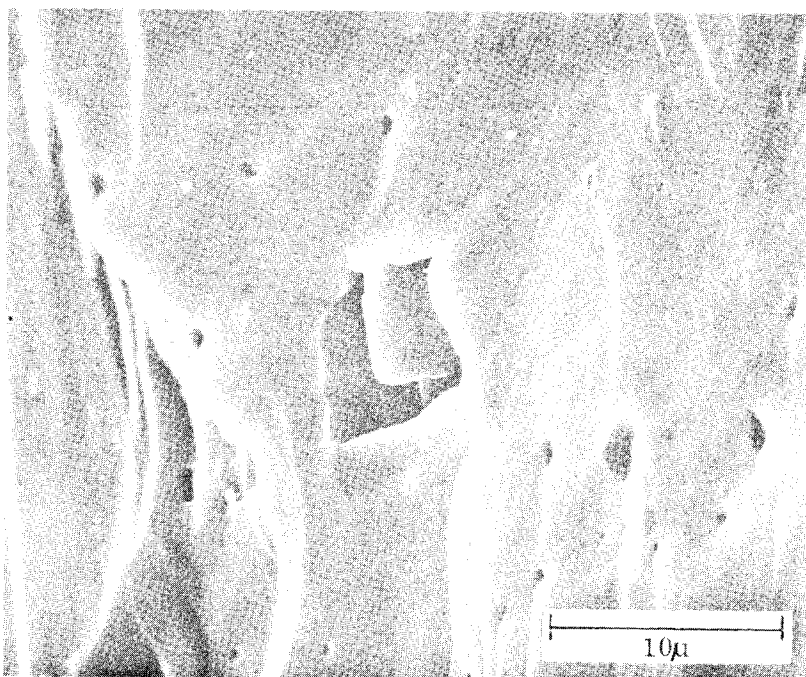


Figure 7. Detail view of pore in center of Figure 6, 3,000 x, note absence of any further indication of micro-roughness.

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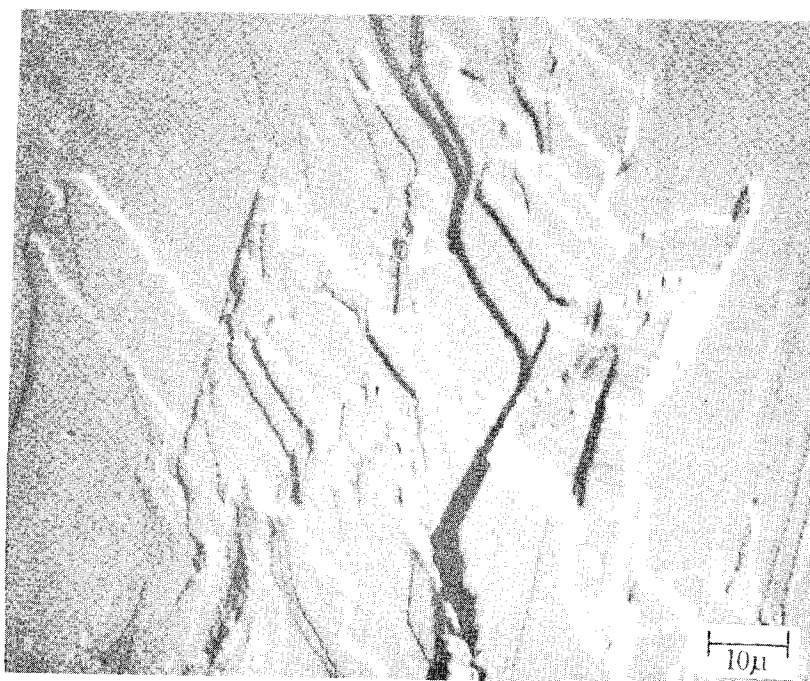


Figure 8. Surface of albite, 1,000 x, showing smooth planar areas interrupted by escarpments perpendicular to the cleavage plane.

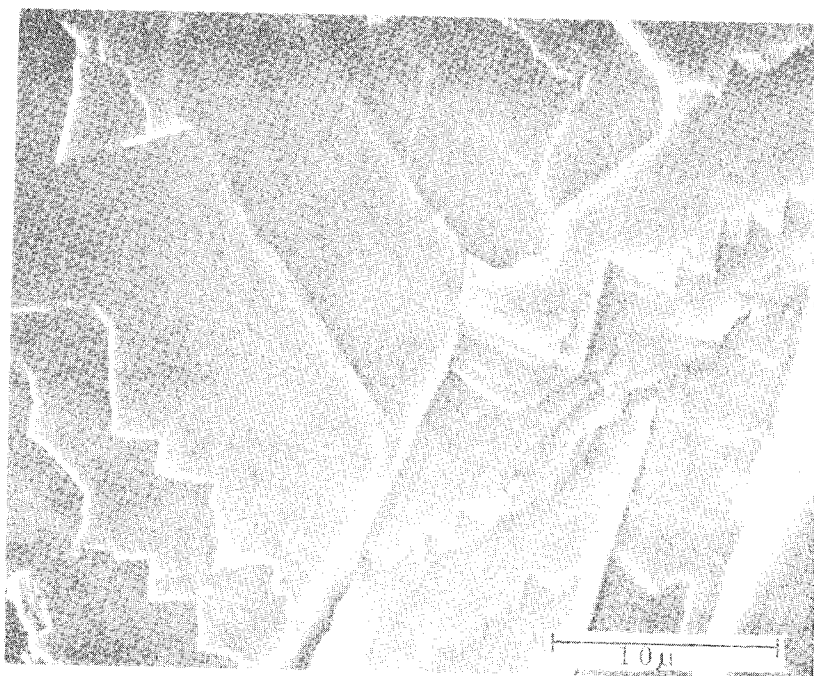


Figure 9. Detail view of central portion of Figure 8, 3,000 x.

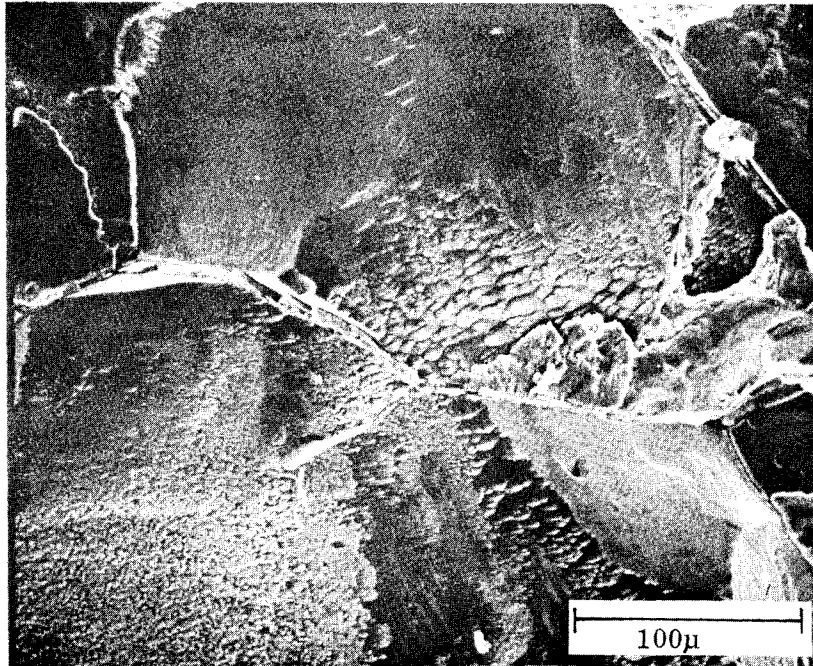


Figure 10. Surface of olivine, 300 x, showing smooth planar areas and areas of rough microcrystalline habit.

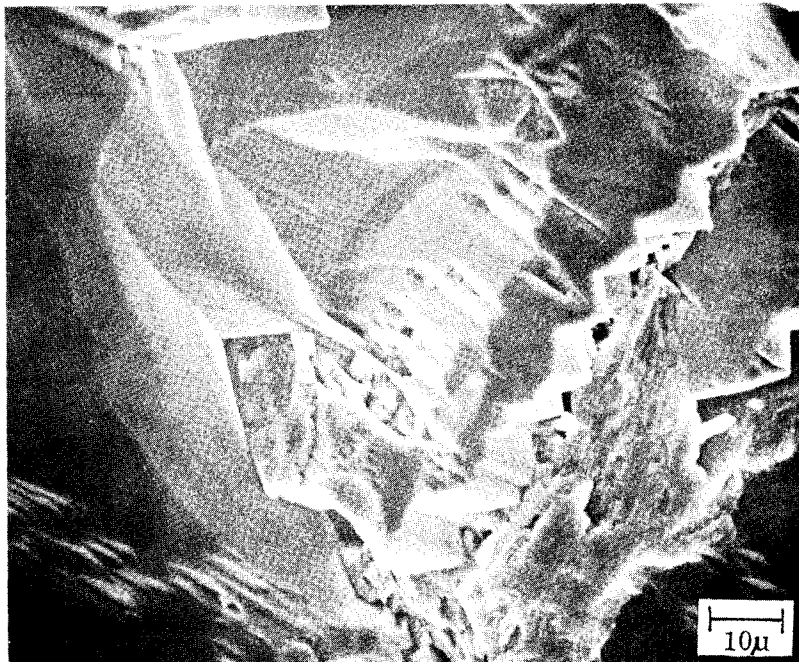


Figure 11. Surface of olivine, 1,000 x, showing microporous structure and view of "dimples" perpendicular to that shown in Figure 10.

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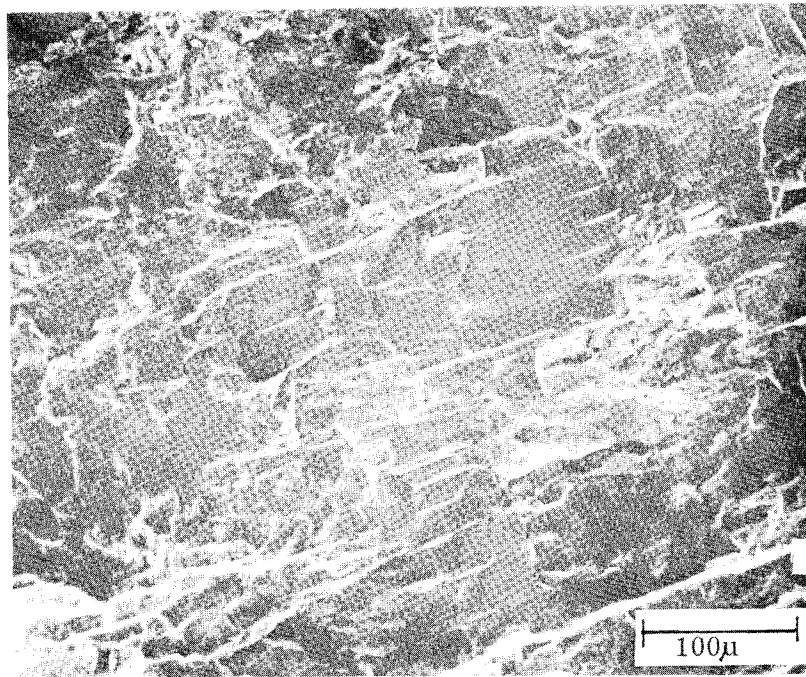


Figure 12. Augite, 200 x, typical view of surface showing cleavage plane and irregularly broken areas of higher relief perpendicular to the cleavage plane.

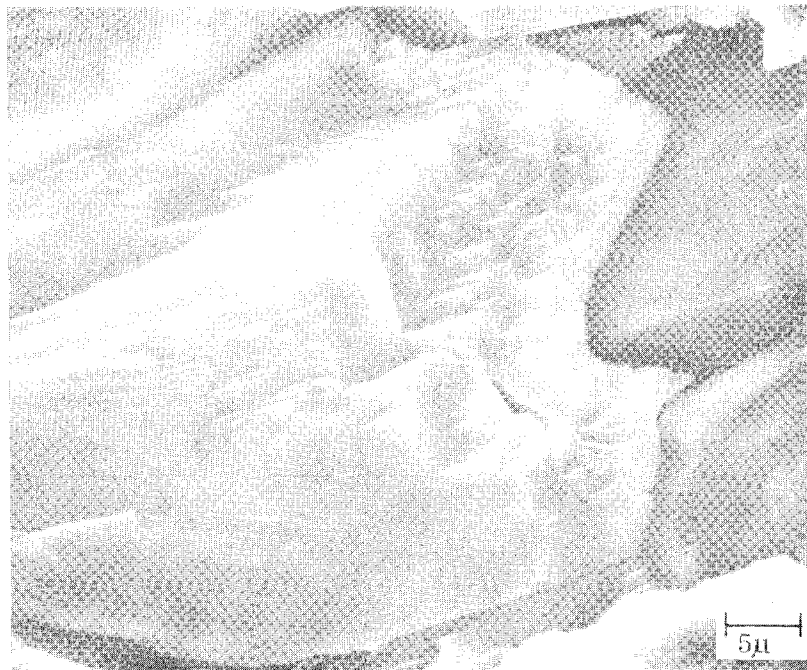


Figure 13. Augite, 2,000 x, showing small area of intersecting cleavage planes rich in microrelief.

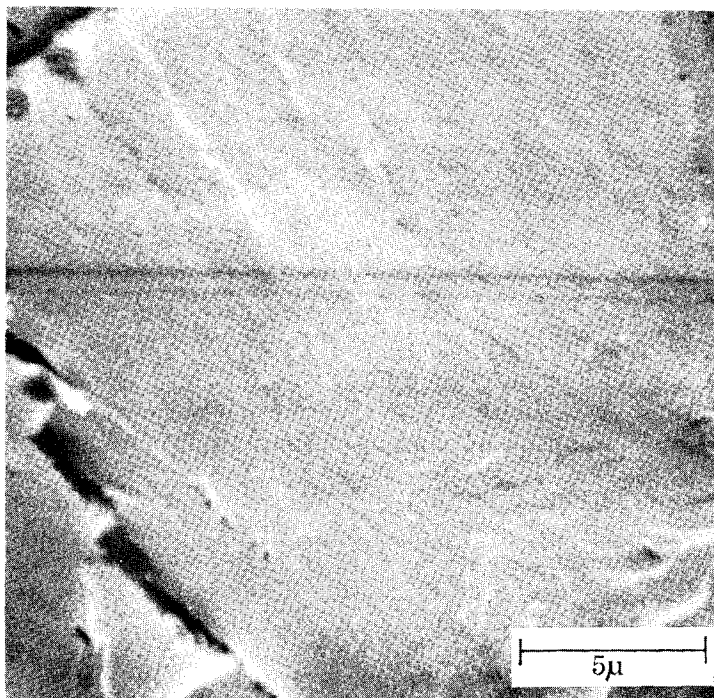


Figure 14. Microcline, 5,000 x, showing smooth surface topography on fracture surface parallel to a cleavage direction.

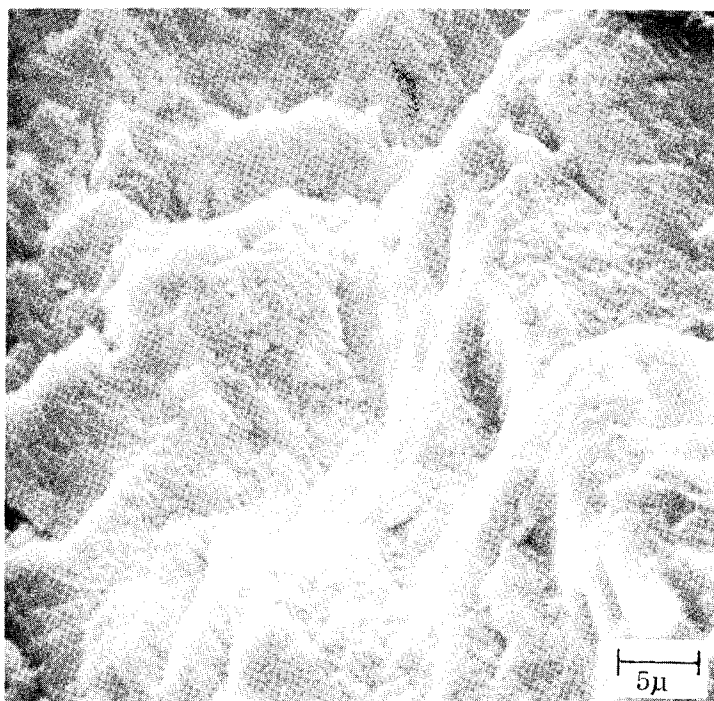


Figure 15. Microcline, 2,000 x, fracture surface showing rough intricate topography.

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Figure 16. Microcline, 2,000 x, showing boundary between two types of surface morphologies shown in Figures 14 and 15. This micrograph represents views parallel to and perpendicular to cleavage.

#### Discussion of Relationship of Surface Roughness to Tensile Strength

It is evident that the intrinsic cleavage of fracture of the mineral is expressed on the microscopic and submicroscopic scale, and that it creates roughness down to the smallest area. There are regions which though smooth on a megascopic scale are extremely rough and craggy on a minute scale and possess many times the apparent, or geometric, surface area.

Of those minerals whose bond strength to cement paste was successfully tested, and for which scanning electron micrographs were taken, quartz was the strongest, followed by dolomite and augite.

It is difficult to assess the roughness factor visually but it would appear on the basis of comparison of the electron micrographs and the amount of area each particular type of surface is estimated to represent that the (descending) order of roughness factors is dolomite, augite, and quartz. The observation that quartz may have relatively

less surface area available for adhesion and still form the strongest bond may be due to pozzolanic action since quartz is entirely silica. At least qualitatively it would appear that dolomite, which forms a stronger bond than augite, does have more available surface area than augite and at the same time has essentially no silica, while augite is a silicate mineral.

## SUMMARY AND CONCLUSIONS

The methods of specimen preparation and tensile testing devised in this investigation allow for the specimen to be continually saturated with water from the time of casting to the moment of failure in the testing machine. Physically and mechanically the procedure is excellent. At its best the preparation and testing worked exactly as planned with specimens having to be discarded only because of defects, cleavages or partings, within the rock disc.

The procedure needs refinement with respect to the following points:

- (1) With certain samples a very limited number of cores was obtained from the sample block during the drilling operation because the paste disbonded from the mineral disc when the core drill encountered either the upper or lower interface with the paste.
- (2) With certain samples, the dispersion of tensile strength values about the average is judged to be too great, and the results do not have a good normal distribution.

Regarding (1), the fact that a large number of test cores had been obtained with limestone, quartz, dolomitic marble, and cement paste indicates that the procedure works, but that some samples may be lost because of variations in other materials, sample preparation, and mixing and drilling techniques. Each step in the procedure needs to be critically evaluated by preparing companion sample blocks: One with a rock or mineral known to have given good results and one with a material failing in preparation. Alternatively, it is possible that the bond strength of those materials that broke in preparation may, in fact, be too low at 27 days to be cored for tensile testing.

With regard to point (2), the variation may be due to the variability in the specimen block caused by the inherent variability of the rock or mineral slice, or of the cement paste, testing procedure, etc. The observed coefficients of variation for the bond test results, however, are not considered excessive in comparison to those found by others (Scholer 1964). In cases such as this, if the variation is inherent and cannot be eliminated, more samples are required to be tested in order to improve

the confidence level of the average value. An advantage of the method developed in this investigation is that large numbers of samples can be prepared economically and conveniently.

The differential capacity method has been identified as being a very promising technique for the determination of roughness factors of mineral surfaces. This method is used in electrochemical research for the purpose of determining the true surface area values of electrodes.



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