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# The Determination of the Crystallite Size in Alumina with Submicron-sized Crystallites

F. K. van Dijen

LONZA-Werke GmbH, Konstanzerstraße 15, 79761 Waldshut-Tiengen, Germany

K. Kampfer & H. Hofmann

Alusuisse-LONZA Services AG, Bad. Bahnhofstraße 16, 8212 Neuhausen am Rheinfall, Switzerland

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***Abstract***

*Sample preparation for SEM-analyses of ceramic microstructures discussed. It is shown that for a submicrometer alumina sample, preparation of polished sections is of utmost importance in order to determine the size of the crystallites correctly.*

*On traite de la préparation des échantillons pour l'analyse au MEB de la microtructure de céramiques. On montre, dans le cas d'un échantillon d'alumine de taille de grains inférieure au micron que la préparation des sections et leur polissage sont cruciaux si l'on veut déterminer correctement la taille des cristallites.*

*Im folgenden wird die Präparation von Keramikgefügen für die SEM-Analyse diskutiert. Es wird gezeigt, daß die Herstellung von polierten Querschnitten zur richtigen Bestimmung der Kristallitgröße im submikrometer Bereich von entscheidender Bedeutung ist.*

## 1 Introduction

Nanophase or submicrometer materials are studied extensively in laboratories. One of the well-known applications of such a material is as abrasive grits, the so called sol–gel abrasive grits from 3M or Norton. Much has been published on the synthesis of this fine grained alumina by the team of Messing and co-workers.[1–3] More information on fine-grained alumina is available from the literature, especially patents.[4–16] One of the crucial characteristics of such a material is the size of the crystallites. This is why the correct determination of the crystallite size is of the utmost importance.

The size of the crystallites is determined with both scanning (SEM) and transmission electron microscopy. With SEM, either a fracture surface or a surface after grinding, polishing and etching is examined.[1–5] The analyses of a fracture surface has the advantage that sample preparation is very simple. However, from the literature on stereology it is known that looking at fracture surfaces does not represent a real microstructure of the material.[17–19] According to Russ: 'One problem with looking at fracture surfaces in materials is that they may not represent valid "random" samples of the material structure.'[18] In this paper we show that the differences in sample preparation result in large differences in the determination of the crystallite size of submicrometer alumina. It will be shown that the analyses of fracture surfaces does not result in the correct size of the crystallites, as can be expected from the literature.[17–19] The analyses of a surface after grinding, polishing and etching may result in the correct determination of the size of the crystallites.

## 2 Experimental

Sintered abrasive grits from Norton, 3M and A-L were analysed. The Norton grits were taken from a grinding wheel. The Norton and 3M grits are made by a sol–gel process. The A-L grits were made by a non sol–gel process.[20] The Norton grits are 99·9% alumina, the A-L grits are 99% alumina and the 3M grits are alumina with rare earth oxides. From the grits the fracture surface was analysed with SEM. The grits were also ground and polished using diamond pastes. These grits were thermally etched in air at 1350°C for 20 min. After etching the surface was analysed with SEM.

The micrographs were manually analysed using the method of the 'mean intercept length'.[2] From each sample at least three micrographs from different grits were analysed. Lines were drawn across the micrographs, assuring that at least 1000 grains were covered by the lines. The mean intercept length, $L$, is calculated according to eqn (1):[19]

$$L = L_0/N \quad (1)$$

with $N$ as the number of crystallites cut by the lines with total length $L_0$[20]. By assuming uniform, spherical crystallites, the diameter of these crystallites, D, is calculated according to eqn (2):[17–19]

$$D = 1{\cdot}5\ L \quad (2)$$

By assuming uniform, equiaxed, non-spherical crystallites, the mean tangent diameter $D_t$, is calculated according to eqn (3):[18]

$$D_t = 1{\cdot}08\ D = 1{\cdot}62\ L \quad (3)$$

As the grits from 3M show a microstructure with plate-like crystallites the authors tried to estimate the average diameter and thickness of these platelets. This was done by optical selection of a few platelets and measuring diameter and thickness.

Exner and Hougardy suggested that the standard deviation s decreases when $N$ increases according to $s \sim N^{-1/2}$.[19] So, accuracy should increase with increasing value of $N$: the more crystallites cut by the lines the higher the accuracy. The accuracy of the determination of the size of the crystallites depends also, however, on the accuracy of counting the number of crystallites cut by the lines. As shown by Russ, a failure in counting can easily occur.[18] We estimate to have an accuracy of about 5%. This indicates that counting 50–100 crystallites may be sufficient to match the accuracy permitted by the risk of miscounts.

## 3 Results

Figures 1, 2 and 3 show the fracture surfaces of Norton, 3M and A-L abrasive grits. These figures seem to indicate that the size of the crystallites ($D_t$) is about out 0·33 $\mu$m for Norton, < 1 $\mu$m for

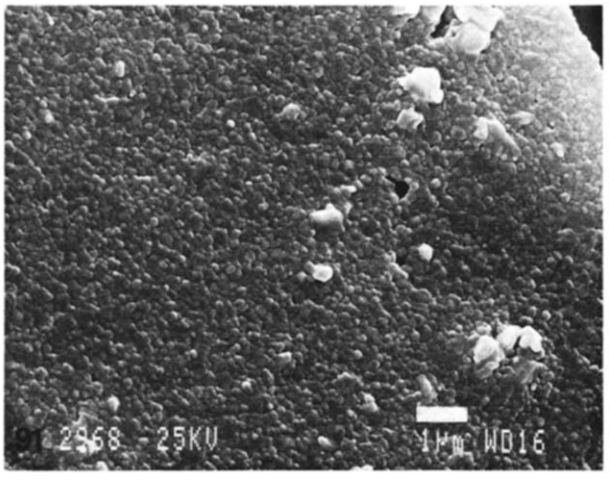


**Fig. 1.** Electron micrograph of a fracture surface of Norton sol–gel abrasive grits. The length of the bar is 1 $\mu$m.

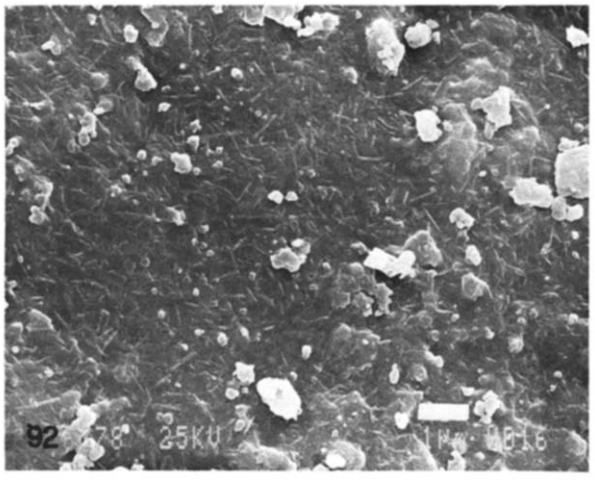


**Fig. 2.** Electron micrograph of a fracture surface of 3M sol–gel abrasive grits. The length of the bar is 1 $\mu$m.

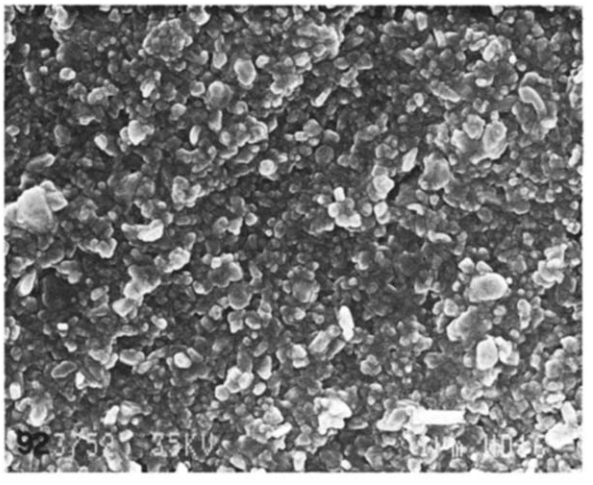

**Fig. 3.** Electron micrograph of a fracture surface of A-L abrasive grits. The length of the bar is 1 $\mu$m.

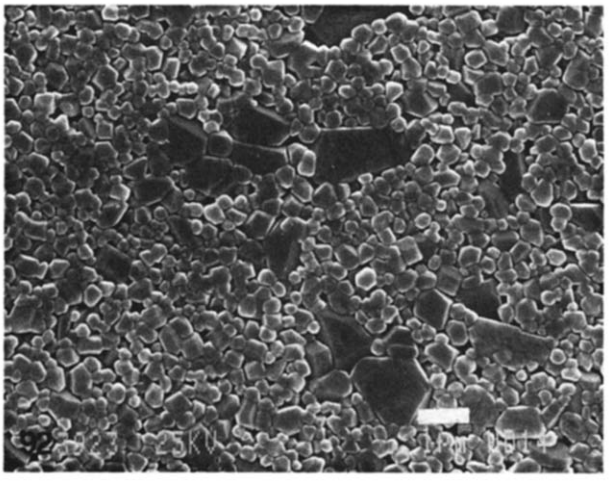

**Fig. 4.** Electron micrograph of a surface after grinding, polishing and etching of Norton sol–gel abrasive grits. The length of the bar is 1 $\mu$m.

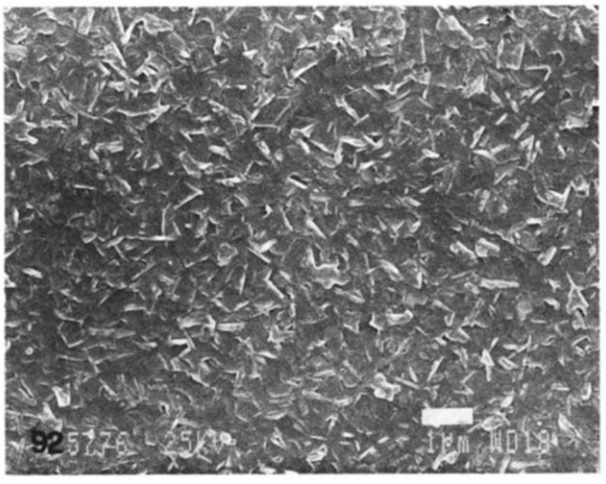

**Fig. 5.** Electron micrograph of a surface after grinding, polishing and etching of 3M sol–gel abrasive grits. The length of the bar is 1 $\mu$m.

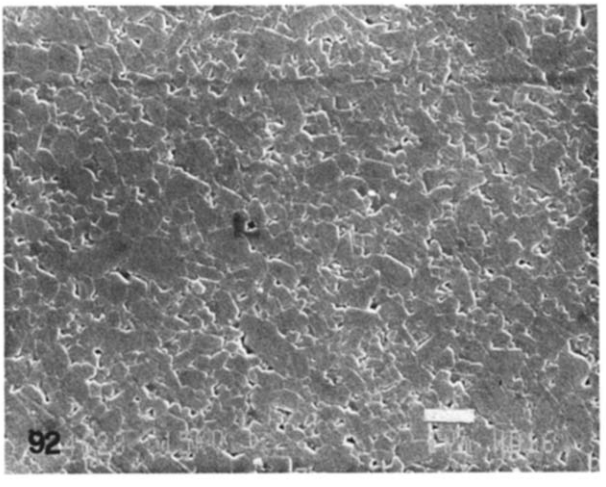

**Fig. 6.** Electron micrograph of a surface after grinding, polishing and etching of A–L abrasive grits. The length of the bar is 1 $\mu$m.

3M and 0.36 $\mu$m for A–L. Figures 4, 5 and 6 show the surfaces of grits from Norton, 3M and A-L after grinding, polishing and thermal etching. These figures indicate using eqn (3) that the size of the crystallites ($D_t$) is about 0·7 $\mu$m for Norton and 0·48 $\mu$m for A–L. The optical selection method led to a value of 0·2 × 1 $\mu$m for 3M. Note the large crystallites in the Norton grits as shown in Fig. 4 and their absence in Fig. 1.

## 4 Discussion

From the results it is clear that looking at a fracture surface suggests crystallites of smaller dimensions than reality. This could be explained by looking at Fig. 7 which shows an intercrystalline fracture surface suggesting smaller crystallite sizes than in reality. In a fracture surface the size of the crystallites suggested is not correct. Even in patents this mistake can be observed, resulting in claims with grain sizes smaller than reality.[8,10]

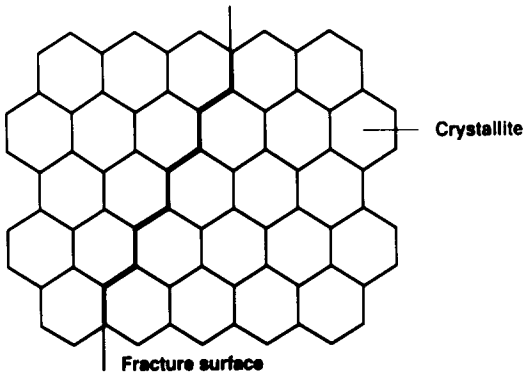


**Fig. 7.** An intercrystalline fracture suggesting crystallites smaller than reality.

It is recommended that fracture surfaces of submicrometer or nanophase materials are not used to determine the size of the crystallites.

## 5 Conclusion

Two methods for analysing the crystallite size in submicrometer or nanophase materials exist, namely fracture surfaces or surfaces after grinding, polishing and etching. Only the latter method shows the correct crystallite size. The fracture surface shows 'crystallites' with smaller dimensions than reality.

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