A.E.R.E.
Harwell

HARWELL

In confidence

The Morphology and Characterization of Talc

B. W. Mott

Solid State Instruments Group,
Materials Development Division,
Building 393,
A.E.R.E., Harwell.

August, 1972

Synopsis

Samples of talc submitted by Johnson and Johnson Ltd. have been examined by a number of physical techniques to characterize the powder and identify any impurities. Examination in both the scanning electron microscope and the transmission electron microscope suggested the possible presence of a few rod-shaped particles as well as the normal platelets but non-dispersive analysis of the X-ray spectra and electron diffraction failed to differentiate between the particles.

X-ray powder diffraction data suggested the presence of a material other than talc in amounts less than 1%. The extra diffraction peaks could not be accounted for completely by published data in the Powder Diffraction File and the most satisfactory conclusion was that the bulk of the impurities were present as Bavalite.

The talc samples supplied to A.E.R.E were of regular Italian talc ex. Val Chisone mine. Grade 00 000

J. R. Dean
Director of Research
Johnson & Johnson U.K Ltd
25th Aug 72

Contents

1. Introduction

Samples of commercially processed talc (magnesium silicate) from their Italian source were received from Johnson and Johnson Ltd. for a thorough characterization together with identification of any natural impurities. After preliminary X-ray diffraction studies had suggested that material other than talc was present at the sub-1% level, samples of chlorite bedrock and magnesite inclusions taken from the Italian mine were supplied as aids to identification.

The various physical methods of examination used were optical microscopy, scanning electron microscopy including non-dispersive analysis, transmission electron microscopy, electron diffraction, X-ray powder diffraction techniques and electron microprobe analysis.

2. Optical Microscopy

A sample of the powder was lightly dusted on to a glass slide and examined under low power binoculars and in a metallurgical microscope. As expected, the majority of the particles were plate-like but a few were observed in the form of rods or needles. In contrast to the majority of the particles which had a whitish lustre, a few were observed with a light green tinge. However, the particle size was too small for adequate resolution optically and it was clearly necessary to resort to electron microscopy to characterize the particles satisfactorily.

3. Scanning Electron Microscopy

3.1 Preparation of Sample

Some powder was dusted on to a specimen stub that had been coated with a colloidal suspension of graphite in alcohol. This gave a thin layer of particles without the formation of large agglommerates. A conducting layer of carbon was subsequently deposited by evaporation on the particles.

3.2 Topographical examination

Examination in the Stereoscan at magnifications up to about x 10,000 generally confirmed the results of the optical work. Platelets were predominantly present in various orientations and some needle-like configurations were clearly accounted for by plates which were viewed in an 'edge-on' position and this was confirmed by taking stereographic photographs. Typical structures obtained are illustrated in Figures 1, 2 and 3.

3.3 Chemical analysis using Non-Dispersive Techniques

Non-dispersive X-ray analysis was carried out on a number of particles using a lithium drifted silicon solid state detector. No significant

variation was found in relative intensity of the silicon and magnesium characteristic emissions suggesting that they were all of similar relative composition. This does not, however, prove that all the particles analysed were talc for the following reasons:- i) there is no guarantee that all the X-ray emission measured was from an individual platelet and ii) it does not exclude the presence of an impurity of similar magnesium/aluminium ratio to that of talc. The X-ray spectra obtained did not show significant emissions of other metallic elements indicating the absence of any major impurity.

4. Transmission electron microscopy

4.1 Preparation of sample

An equal volume of talc powder and a solution of 15% nitrocellulose in amyl acetate was rubbed on to a glass plate with a spatula. This aliquot was then diluted with acetone and spread over a clean microscope slide to dry. The dried film was scored into 3mm square sections which were floated off the slide on to water and picked up with an electron microscope grid. After drying off on a filter paper, a supporting film was then evaporated over the surface. This yielded a nitrocellulose film in which the talc platelets were encapsulated with their planes lying generally in that of the film to preclude 'edge-on' effects.

4.2 Morphology

The platelets in the film were examined in an EM300 transmission microscope at magnifications of up to x 80,000. This revealed that the rod-shaped particles which were about 250 Å diameter and varied in length up to 0.15mμ, could have been platelets which had folded or rolled up, but this was not positively confirmed (see figure 4).

4.3 Electron diffraction

Electron diffraction patterns were obtained in situ in the microscope from a number of particles with both types of appearance but no real evidence was obtained of a structural difference between the plates and "rods". Two diffraction patterns showed evidence of preferred orientation but this was to be expected from their nature and was not considered to be indicative of any chemical difference between the two particles. It must be noted, however, that although the method of preparing the sample for examination had been chosen to separate the particles as much as possible, there was difficulty in guaranteeing that completely isolated particles had been selected for diffraction.

5. X-ray diffraction

5.1 Experimental

Some of the powder was packed in silica capillary tubes for both X-ray ...er diffraction patterns and diffractometer traces. The results showed ...nce of preferred orientation and this was probably due to the platelets ...ning themselves in the silica tube when tapped in the usual way to concentrate them in the bottom portion. Patterns were obtained from two completely different batches of talc supplied by the firm and also from two mineral samples said to be Magnesite and Chlorite, although the latter was subsequently discovered to be Muscavite, a micaceous mineral.

The same reflections were obtained from both talc samples but some differences were observed in the relative intensities of some reflections. Since some preferred orientation was evident in both patterns, these intensity differences were most likely due to slight variations in the type and/or degree of preferred orientation of the platelets in the two samples. The overall conclusion was that the two talc samples were identical in nature and that any impurity was due to the same compound or compounds.

The first sixty six reflections from the patterns were selected for identification and the calculated d-spacings are listed in Table I. Of these, all but 27 were satisfactorily accounted for by the available data on talc published in the Powder Diffraction File. This strongly suggested that an impurity was present to an extent judged to be below the 1% level. The strongest reflections from the samples of Magnesite and Muscavite were absent in our talc patterns so that neither could reasonably account for the impurity reflections.

A check was made of the possibility that the impurity was Tremolite but none of the extra reflections agreed with the published data for this mineral. There are a wide range of minerals in the Chlorite group which is a distinct possibility as an impurity since their crystal structure is related to that of talc and brucite. X-ray diffraction data have been published on five varieties and these were checked with the talc patters. In the case of Bavalite (a variety of Daphnite), it was found that 24 of our 66 reflections could be accounted for by this form of Chlorite, although 16 of these were also included in the published data for talc. Bavalite can contain traces of alkaline earths, copper, titanium or manganese but the sample from which the X-ray data were obtained was reported to be $(Mg_{0.4}Fe_{4.2}Al_{1.5})(Si_{2.6}Al_{1.4})O_{10.2}(OH)_{7.8}$.

5.2 Attempts to identify additional reflections

In attempts to prove or disprove that the extra 27 reflections were all accounted for by a small percentage of impurity, a number of approaches were investigated. A thorough computer search of the Powder Diffraction File was made to list possible compounds which could account for the unidentified reflections. Of a whole variety of possibilities including both inorganic and organic compounds, the only three which appeared at all possible were Bavalite, potassium dizirconate $K_2Zr_2O_5$ and calcium aluminium oxide hydrate, $CaAl_2O_4.10H_2O$. In the case of the dizirconate, the talc patterns account for only two low angle lines of medium intensity and other stronger lines are not present. The two strongest lines of the calcium compound could be accounted for by two low angle lines in our patterns plus two others at higher angles (where talc reflections also occur) but that would still leave eleven lines unidentified. On the basis of published X-ray data therefore, Bavalite appeared to be the most likely impurity.

Since both the talc patterns were consistent with some preferred orientation being present in the powder samples, it was considered that some of the extra reflections might have been due to enhancement of very weak reflections from talc which would not be observed in the standard pattern from a perfectly random sample. A computer calculation was made of all possible reflections from talc assuming that the published crystal structure was correct and on this basis, a further 16 of the 27 unknown reflections in the talc patterns could be satisfactorily indexed.

6. Electron Microprobe Analysis

As the X-ray diffraction data strongly suggested the presence of an impurity which could not be identified unambiguously as Bavalite, a limited amount of electron microprobe analysis was considered worthwhile. The sensitivity of this instrument for detecting impurity elements is significantly better than that of the scanning electron microscope and these could have been missed in the work described in Section 3.3. The seven elements selected as being most representative of the likely impurity materials were fluorine, potassium, calcium, nickel, aluminium, iron and chromium. The last four of these might be expected if some varieties of Chlorite were present, calcium for Tremolite, calcium and iron for Actinolite and calcium, fluorine and potassium for Apophyllite. The presence of iron and aluminium would substantiate the possibility of Bavalite.

A sample of the talc was sprinkled on a bakelite disc to give as even a layer as possible and a search was made for each of the seven elements listed. Although the level of the characteristic X-rays for iron was very close to that of the background, there was a possibility that iron was present in a very low concentration throughout the sample. A few discrete areas were found in which calcium and aluminium were detected together as shown in Figures 5 and 6 but the origin of the characteristic X-rays from these two elements could not be traced to individual particles as the lateral and depth resolution of the electron beam was much greater than the average particle size. None of the elements fluorine, potassium, nickel and chromium were present at the level of sensitivity which was about 100 ppm. The results were therefore inconclusive although not inconsistent with the possibility that Bavalite was the major impurity.

7. Discussion

The main evidence for the presence of an impurity in the talc samples comes from the X-ray diffraction data which shows some 27 reflections in addition to those included in the published information. Just over half of these extra reflections can be accounted for by assuming that some weak reflections, not normall of significance for a randomly oriented sample, are enhanced due to preferential type of packing the flakes in the silica tube. This still leaves some reflections completely unaccounted for by talc, the strongest of which agreed with the published data for Bavalite. This mineral is said to occur in the form of sheets and to be greenish in colour which would be consistent with the observations in the optical microscope. Since neither the transmission or scanning electron microscopy was able to distinguish between the rod like particles and the platelets, it seems probable that these were folded platelets as postulated. The limited electron microprobe analysis carried out indicated that calcium and aluminium occurred together at the impurity level in the powder and that traces of iron were also present. This would also be consistent with the presence of a small concentration of Bavalite if some of the magnesium were replaced by calcium as is known to occur in some varieties. Other possible explanations of the extra reflections in our X-ray powder diffraction data are (a) that the published data on mineral talc is incomplete or (b) that small concentrations of impurities such as calcium and aluminium are present in the talc and give rise to the additional lines by slightly modifying the crystal structure. Neither explanation seems acceptable and the possibility of Bavalite being present in an amount less than 1% appears more likely and to fit all the observations.

8. Conclusions

The two samples of talc examined are essentially the same and contain a common impurity present in an amount less than 1%. The extra reflections in the X-ray powder pattern not accounted for by talc are consistent with the impurity being one form of Chlorite known as Bavalite.

9. Acknowledgements

The work reported was carried out by various members of the Solid State Instruments Group whose suggestions and comments are greatly appreciated.

Circulation

Messrs. Johnson and Johnson Ltd. (5 copies)
Commercial Office
A.E.R.E. Library
Mr. B. W. Mott

Table I X-Ray Powder Diffraction Data

	Data from Diffractometer Trace		Data from Powder Diffraction File			
			P.D.F. 19-770 Talc		P.D.F. 7-166 Bavalite	
	d spacings (observed)	Intensity % of strongest	d	I	d	I
**	13.9846	13			14.0	60
	9.3585	100	9.35	100		
**	8.6130	31				
**	8.1539	7				
**	7.4367	2				
**	7.0418	44			7.08	100
**	6.5298	1				
	4.6756	84	4.67	8	4.681	30
	4.5428	11	4.56	25		
	4.3218	8	4.33	6		
*	4.2462	1				
	4.1235	3	4.12	6		
	3.8750	1	3.87	2		
	3.5366	49	3.52	< 2	3.523	50
*	3.4386	3				
*	3.3337	36				
*	3.2867	3				
*	3.2456	4				
*	3.1921	20				
	3.1125	89	3.12	40		
*	2.8883	37				
*	2.8330	18			2.821	10
**	2.7993	1				
*	2.7279	4				
	2.6254	2	2.627	8	2.619	30
	2.5887	5	2.589	14	2.574	20
*	2.5321	1				
	2.4747	10	2.479	30	2.469	10
*	2.4200	2			2.405	20
	2.3394	36	2.337	2		
	2.2769	1	2.289	2	2.279	10
	2.2116	3	2.219	6		
	2.1910	3	2.188	2		
	2.0987	1	2.086	2		
	2.0668	1	2.071	2		

	Data from Diffractometer Trace		Data from Powder Diffraction File			
			P.D.F. 19-770 Talc		P.D.F. 7-166 Bavalite	
	d spacings (observed)	Intensity % of strongest	d	I	d	I
*	2.0281	17			2.014	20
*	1.9997	1				
	1.9786	1	1.967	2		
	1.9179	1	1.921	2	1.893	5
	1.8697	71	1.870	4		
*	1.8173	1			1.828	5
*	1.7857	1			1.765	5
	1.7338	2	1.731	10	1.722	5
	1.6879	7	1.684	6		
	1.6715	8	1.668	6	1.666	5
	1.6529	4	1.654	2		
*	1.5777	1			1.560	20
	1.5573	56	1.558	2		
	1.5418	1	1.540	< 2		
	1.5268	4	1.529	55	1.523	10
**	1.4764	1			1.483	5
**	1.4650	2				
**	1.4473	2				
**	1.4338	5			1.427	5
	1.4145	7	1.411	4	1.411	5
	1.3958	12	1.386	4	1.393	5
	1.3858	12	1.376	2		
	1.3354	48	1.336	2	1.339	5
	1.3179	1	1.318	8		
	1.2945	2	1.295	10		
	1.2500	1	1.255	2		
	1.2411	2	1.243	2		
	1.2383	2	1.233	2		
	1.2230	6	1.219	< 2	1.228	5
	1.1828	1	1.187	4		
	1.1688	10	1.169	2		

* Accountable on computer-calculated pattern for TALC
** Unaccountable on basis of TALC
N.B. Calculated TALC pattern did not extend below d = 1.600

FIGURE 1. Scanning Electron Micrograph x 500

FIGURE 2. Scanning Electron Micrograph x 500

FIGURE 3. Scanning Electron Micrograph x 10,000

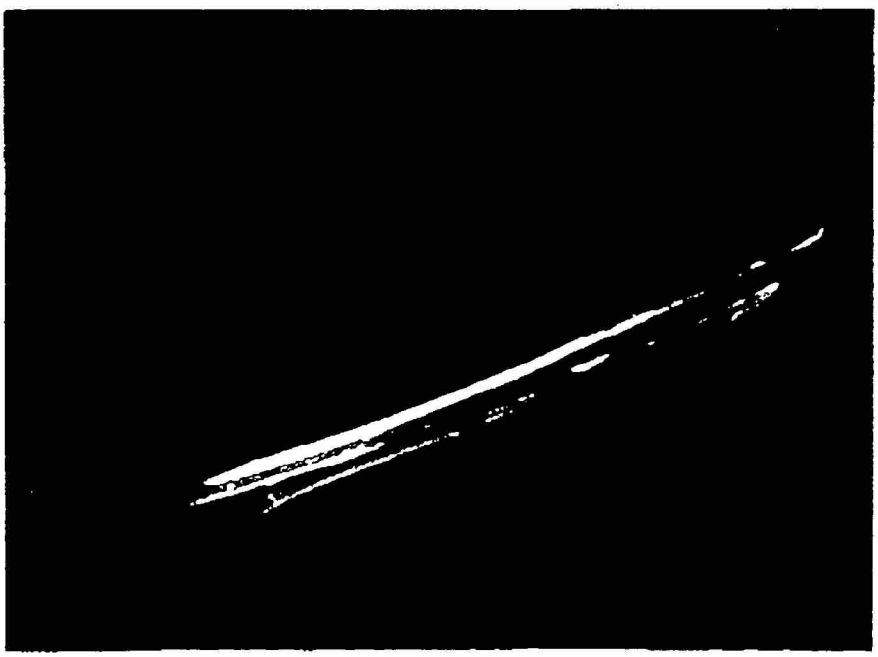

FIGURE 4. Transmission Electron Micrograph x 10,000

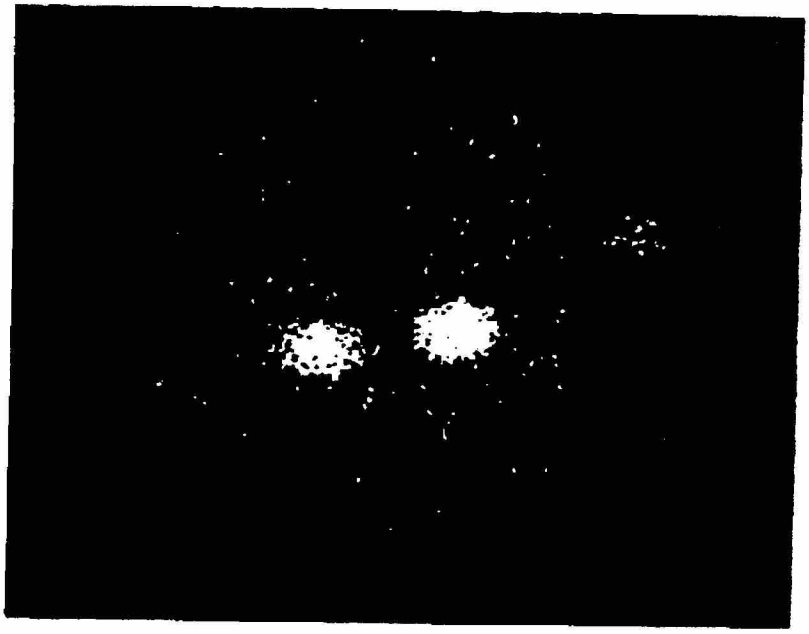

FIGURE 5. Electron Microprobe Scan for Calcium

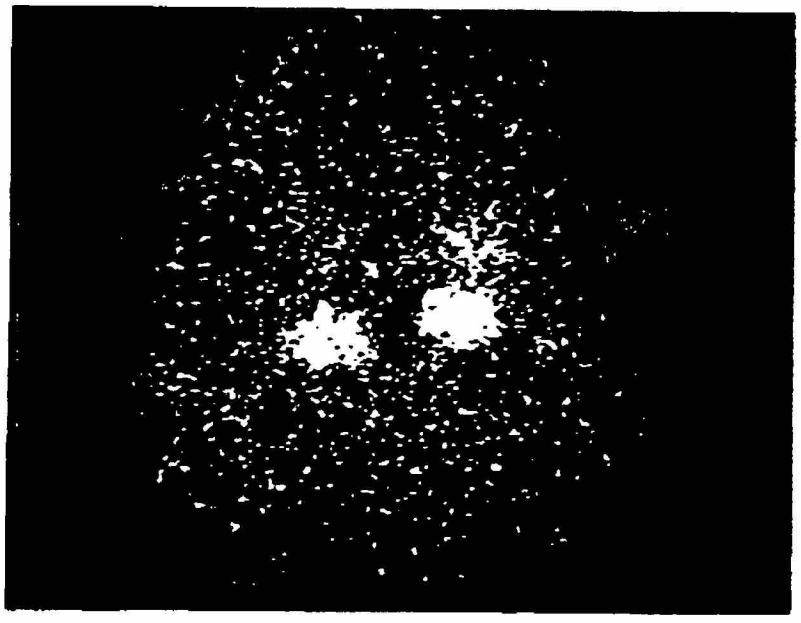

FIGURE 6. Electron Microprobe Scan for Aluminium in Same Area as Above