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TOPAZ RESEARCH PROJECT - POTENTIAL TRAJECTORIES

LATE-BREAKING DEVELOPMENTS

- Proof-of-concept for templated mullite fibre growth achieved
- Potential for mullite fibre growth without templating indicated
- Unexpected observation of extractable single-crystal alumina platelets made
- Potential for mullite-reinforced composite fabrication established
- Feasibility of topaz-quartz quantification and isolation demonstrated

Background

Topfibre Pty. Ltd. (a wholly owned subsidiary of ASX listed Toptung Limited) is undertaking a collaborative research programme in partnership with the School of Materials Science and Engineering, UNSW Sydney (Sydney, Australia). The project is led by Prof. C.C. Sorrell, who has >20 years' experience working on the conversion of topaz to mullite ($3Al_2O_3{\cdot}2SiO_2$). The research programme is funded principally through an Australian Research Council Linkage Grant, which has tenure of 3 years (2017-2020) and a total budget of $340,000. The principal aim of the research is to produce on a commercial basis single-crystal, separable, oriented, mullite fibres from topaz concentrate produced from the silexite deposits at the Company's Torrington Project. The research programme targets the (a) development of oriented templates and (b) processing protocols to achieve oriented fibre growth.

The main budget items for this project are (a) one full-time Research Associate and (b) design, construction, and purchase of a specialised furnace with suitable high-temperature capacity and precision pulling rates. The labour requirement was met through the employment of two of Prof. Sorrell's former Ph.D. students (one junior and one senior) and, after some delays, the furnace was commissioned, calibrated, and trialled. These procedures had to be preceded by meeting all of the required technical and safety protocols.

Aligned Mullite Fibre Growth

Figure 1 below shows proof-of-concept that separate and aligned single-crystal fibres can be produced by the proposed templating method. The image shows that the aligned template at the bottom of the figure facilitates the initial growth of mullite whiskers, which will be coarsened into fibres during the progress of the heating process.

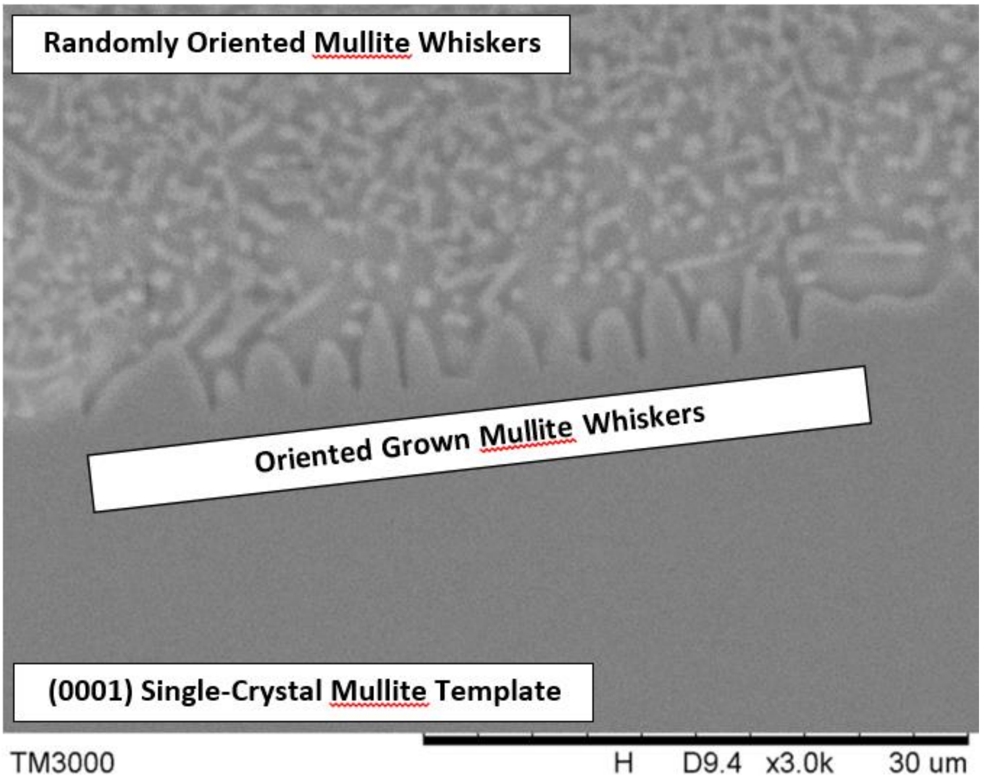

Figure 1: Interface between aligned (0001) single-crystal mullite and mullite from calcined topaz

Unconstrained Mullite Fibre Growth

Figure 2 below shows that it may not be required to grow aligned fibres as it may be possible to design the processing so that fully grown fibres can be generated in a felted mass in which the fibres are weakly bonded and hence mechanically separable.

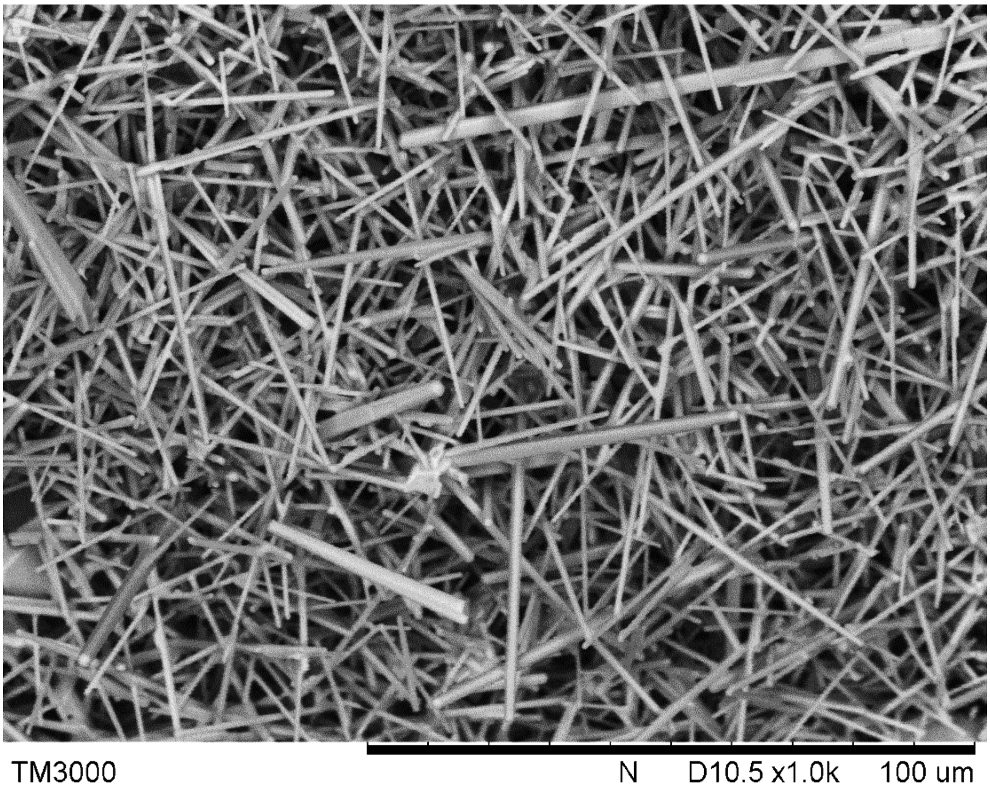

Figure 2: Felted mullite whisker mass from topaz ball milled for 196 h and calcined for 2 h at 1600°C

This image above reveals few of the universally observed intergrowths in typical mullite fibre arrays, which make separation without fracture impossible. Here, it appears that the whiskers form only point contacts, which may make it possible to separate the fibres without breakage. Preliminary attempts to separate the fibres suggest that they are insufficiently calcined to allow separation since they remain mutually attached by incompletely calcined topaz.

Alumina Platelet Formation

Previous work by Prof. Sorrell revealed that single-crystal alumina (Al_2O_3) could be produced from the calcination of topaz when zirconia (ZrO_2) was added to the topaz. Surprisingly, Figure 3 shows that these platelets formed from topaz alone, albeit when fired at more modest conditions than normally used.

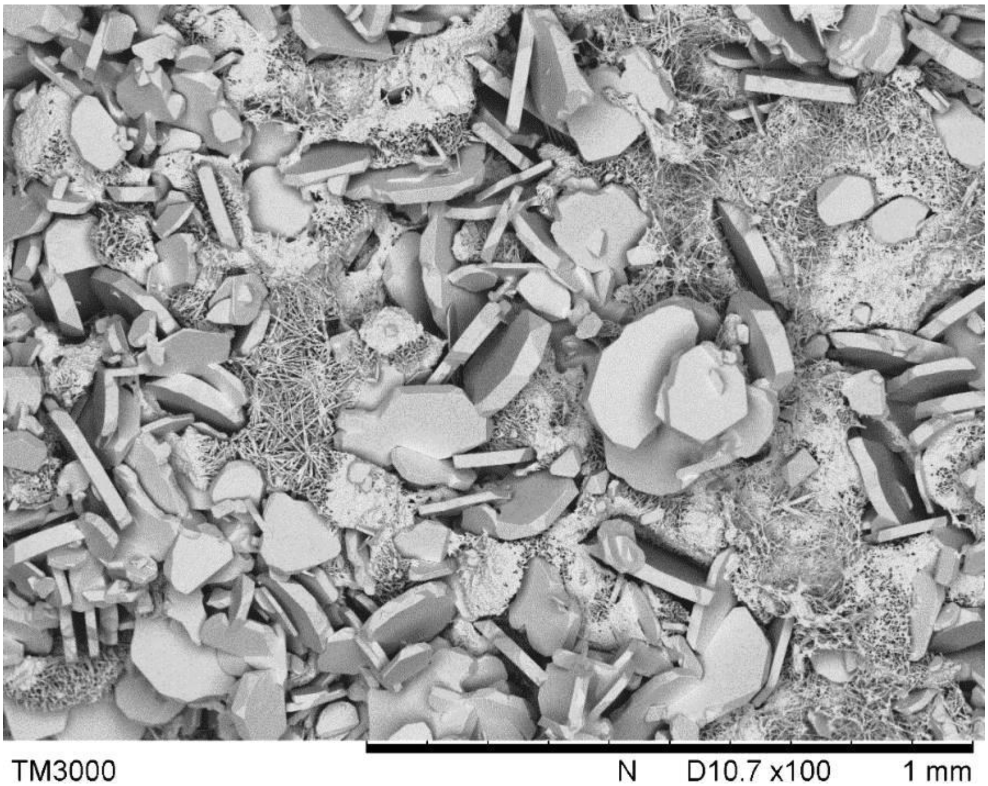

Figure 3: Single-crystal alumina platelets from topaz ball milled for 48 h and calcined for 2 h at 1600°C

This image reveals that the large alumina platelets exhibit few intergrowths and very poor bonding to the surrounding mullite whisker matrix. These observations suggest that it may be relatively easy to isolate the platelets from themselves and the matrix.

Metal-Infiltrated Metal Matrix Composite Fabrication

Figure 2 above shows that it is possible to produce continuously interlocked mullite fibre compacts, the bonding of which can be increased by heat treatment. The capacity to produce a rigid single-crystal felted mass presents a unique preform for vacuum infiltration by molten metals, such as aluminium. Figure 4 illustrates a 3D schematic of the outcome of this process.

A grant application for $100,000 funding for 6 months' tenure has been submitted to the newly established Defence Innovation Network (DIN) in order to demonstrate proof-of-concept.

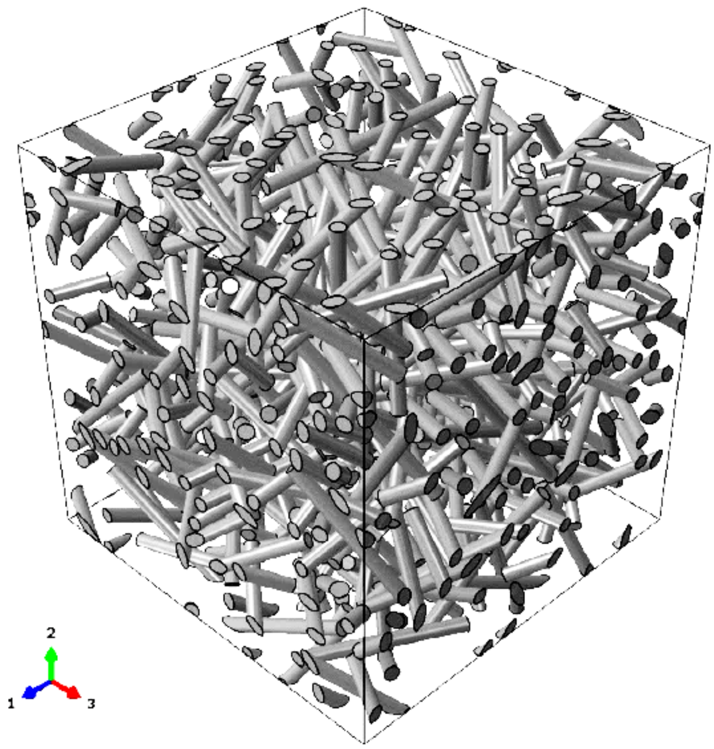

Figure 4: Schematic of 3D porous mullite fibres compact suitable for metal matrix composites

Quantification of Topaz-Quartz Mixtures

The potential to achieve rapid commercialisation of topaz as an abrasive is hindered by the presence of α-quartz (α-SiO_2), which is known to cause silicosis when inhaled, particularly when freshly crushed. It often is difficult to determine the amount of α-quartz in mixtures owing to its presence at low levels and the limited sensitivity of the relevant analytical equipment. Figure 5 shows that the topaz has α-quartz levels sufficiently high to be detected by X-ray diffraction. Consequently, a series of standards with known low α-quartz levels can be prepared and compared with results obtained from quantitative analytical software. This process will result in (a) development of a rapid and inexpensive method of determination of α-quartz content and (b) determination of the limit of resolution (in terms of α-quartz content) of the X-ray diffraction method.

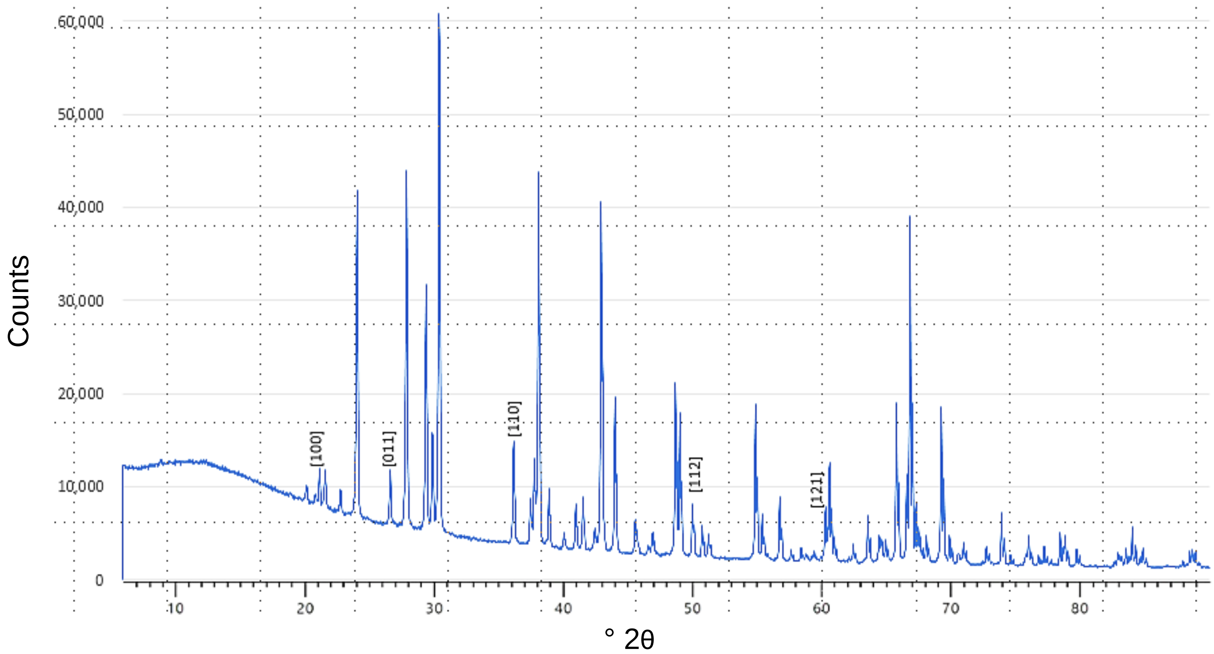

Figure 5. X-ray diffraction pattern of topaz (unambiguous indexed peaks are α-quartz)

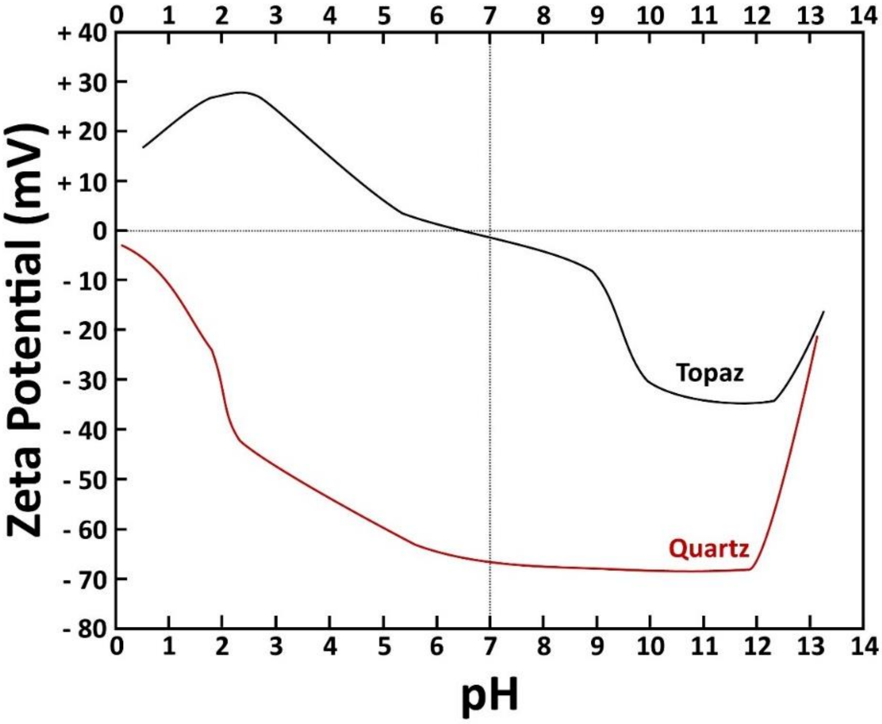

Figure 6: Zeta potentials of topaz and quartz as a function of pH

These data reveal that the use of neutral pH (*viz.*, water) or a slightly acidic solution (*e.g.*, with added vinegar) is suitable for the separation of the two minerals. However, since the particles are large, they must be suspended using a charging agent, which also will allow even greater capacity to facilitate mutual repulsion.

Commercialisation Directions (Mining, Aerospace, and Military Industries) is as follows:

1) Fabrication of individual mullite fibres for metal matrix composites (MMCs) and ceramic matrix composites (CMCs)
2) Fabrication of mullite preforms for MMCs
3) Fabrication of alumina platelets for MMCs and CMCs
4) Purification of raw topaz for the abrasives industry

Research Directions

The nature of the ARC Linkage Grant Application is such that deviations from the proposed direction are possible as long as the overall goal remains the focus. Consequently, considerable flexibility in research directions is possible:

Mullite Fibres for MMCs and CMCs

This is the main thrust of the current research programme. Proof-of-concept of the templating technique has been demonstrated. However, it is possible that this may not be required if (a) the whiskers can be coarsened to fibres without causing intergrowth and (b) the fibres can be separated without excessive breakage. Both streams of work represent the main foci of the current research.

Mullite Preforms for MMCs

Regardless of success or failure of the DIN grant application, this work will proceed. This is the case because (a) Prof. Sorrell's colleagues in the School of Materials Science and Engineering, Prof. M. Ferry and Dr. K. Laws, are experienced in vacuum infiltration of molten metals. (b) they have the requisite facilities to do the work, and (c) the probability of producing a commercial product in a short time frame is high.

Alumina Platelets for MMCs and CMCs

As there is an established market for alumina platelets, which are relatively expensive, this research is underway. The goals are to (a) produce the alumina platelets at the minimal temperature and time possible so that they are separable from the matrix, (b) determine the minimal temperature and time required to generate platelets of suitable size, and (c) determine the conditions suitable to maximise the proportion of platelets.

Raw Topaz for Abrasives

As there is an established market for topaz as an abrasive, this research is underway. The goals are to (a) trial three suitable charging agents, (b) determine the conditions necessary for particle suspension, (c) determine the pH range suitable for the preceding, (d) separate the particles by sedimentation (topaz sinking, α-quartz floating), and (e) apply the developed technique of quantitative analysis.

Other Comments

1) Although not critical, the current work confirms that a powder bed (rather than separated particles) of topaz is required to maintain the gas phase during processing.
2) The gas phase is a critical issue because it contains silicon tetrafluoride (SiF_4), which is highly toxic. So the processing must be carefully designed and implemented.
3) Since whiskers are a health risk owing to the potential for inhalation and the consequent potential to develop mesothelioma, these represent a second driving force to ensure that safety is a top priority.
4) The processes to produce mullite fibres, mullite preforms, and alumina platelets can be scaled up with the use of an induction furnace with a large coil. A large topaz compact can be pulled through the hot zone, thereby converting the powder mass accordingly. Continuous maintenance of the topaz compact by periodic stacking will allow the process to be done continuously.
5) The target fibre diameter and length are 12 μm and 35 μm, respectively, so coarsening of the whiskers is required although lengthening will not be an issue. These requirements may impact on the feasibility of achieving unconstrained fibre growth.
6) The variables available to achieve the preceding are temperature, time, and milling (*i.e.*, particle size).

For, and on behalf of, the Board of Directors of TopTung Limited,

Dr Leon Pretorius

Executive Chairman

TopTung Limited

25 July 2018

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