Original Paper

Development of a soluble high-temperature insulation fibre

Iain C. Alexander and Gary A. Jubb

Morgan Materials Technology Ltd., Stourport-on-Severn (UK)

The three variables, dose, dimensions and durability, have been shown from many studies to be the factors that combine to give the potential for a fibre to cause respiratory diseases. Considering durability, a fibre that could show increased dissolution, possibly accompanied by chemical change, in physiological solutions would be expected to show a reduced persistence in the lungs and have a lower potential for producing respiratory diseases. The development of such a fibre, Superwool X607, and the understanding that has been acquired in terms of solubility rate and other fibre properties is presented in this paper.

The importance of in-vitro solubility rate studies to screen potentially useful fibre compositions, combined with an understanding of how these compositions can be selected using free energy of hydration or non-bridging oxygen theories, is discussed in terms of developing new less "in-vivo" durable fibres.

Entwicklung einer löslichen Isolierfaser für hohe Temperaturen

In zahlreichen Studien ist aufgezeigt worden, daß für eine Faser die drei Variablen Dosis, Abmessungen und chemische Beständigkeit in der Kombination die Faktoren darstellen, die Atemwegserkrankungen auslösen können. Betrachtet man die Beständigkeit, so kann festgestellt werden, daß eine Faser mit erhöhter Löslichkeit in physiologischen Lösungen (möglicherweise hervorgerufen durch eine chemische Veränderung) auch eine verringerte Persistenz in den Lungen zeigt und damit ein geringeres Potential für das Entstehen von Atemwegserkrankungen besitzt. In der vorliegenden Arbeit werden die Entwicklung einer entsprechenden Faser, Superwool X607, und die in Form von Auflösungsgeschwindigkeit und weiteren Fasereigenschaften verfügbaren Erkenntnisse vorgestellt.

Die Bedeutung von Untersuchungen der In-vitro-Auflösungsgeschwindigkeit zum Auffinden potentiell verwendbarer Faserzusammensetzungen wird mit Hilfe der Theorie der freien Hydratationsenergie oder der Theorie nichtbrückenbildender Sauerstoffe erörtert, um ein Verständnis für die Auswahl der Glaszusammensetzung zu erhalten und um somit neue Fasern mit geringerer In-vivo-Beständigkeit zu entwickeln.

1. Introduction

Only amphibole asbestos and erionite have been shown to be unambiguously pathogenic in man, whereas there is no evidence for man-made vitreous fibres (MMVFs). From a combination of observations in man and from animal experiments in recent years, the potential for fibres to cause respiratory diseases can be related [1 to 3] to three variables, colloquially known as the three 'D's: first, Dose; second, Dimensions; third, Durability.

Clearly as with all materials there is a need for a sufficient dose of fibres, and as with all inhaled materials this must mean the dose of fibre present in the lung. This is related to both the dimension of the fibres and to their residence time in the lung. Thus, the durability or biopersistence of a fibre is a critical determinant of hazard. In fact, it is believed by some, that if the dose of respirable fibres with the key dimensions of diameter $<1\,\mu m$, length $>5\,\mu m$ and aspect ratio $>3:1$ is great enough, then only lack of fibre durability can prevent the development of respiratory disease [4].

Received July 8, 1996, revised manuscript April 7, 1997.

There could be a number of approaches that could be used to reduce the potential for fibrous insulation materials to pose a respiratory hazard, but this paper will concentrate on the concept of producing less durable fibres. The persistence of fibres in the lung, besides being dependent on factors that influence the normal clearance mechanisms (by macrophages and lymphatic drainage), is affected by solubility rate, fragmentation and chemical change during the dissolution of the fibre. Vitreous fibres that show some preferential leaching of at least one of the alkali or alkaline earth components of the fibre, and therefore a change in chemical composition, tend to show higher dissolution rates than compositions that show uniform dissolution of all the chemical components of the fibre [5]. Therefore, a fibre that could show increased dissolution, possibly accompanied by chemical change, in physiological solutions would be expected to show a reduced persistence in the lungs and have a lower potential for producing respiratory diseases. This concept has been included in a proposed classification matrix (see table 1) being considered within the European Union by the Commission's DGXI committee [6]. In this a higher content of alkali metal oxide and/or

Table 1. Proposed DGXI (EEC) fibre classification [6]

fibre size: length weighted, geometric mean	fibre matrices with a total content (in wt%) of alkali metal + alkaline earth oxides		
	2 wt%	2 to 18 wt%	18 wt%
coarse: >6 µm	A	D	G bonded mat fibres
standard: 1 to 6 µm	B standard RCF	E	H standard insulation wools
superfine: <1 µm	C superfine RCF	F	I special glass fibres

Explanation: Each box of the matrix (labelled A to I) will be given a hazard classification of 2, 3 or 0; 0 representing no believed cancer hazard.

Table 2. Biocompatible/non-toxic components for a glass forming soluble MMMF

component	general effect on refractoriness	general effect on solubility rate
Al_2O_3	++	−−
ZrO_2	++	−−
TiO_2	+	−
SiO_2	0	0
phosphate	0	+
borate	−−	++
CaO	−	+
MgO	−	+
SrO	−	+
Na_2O	=	++
K_2O	=	++

Explanations: + = positive effect, −: negative effect, 0: no effect.

alkaline earth oxide, which generally gives an increased solubility rate, would lead to a less stringent carcinogen classification of the fibre.

Thus, a reduction in retained lung dose could be achieved, besides by workplace controls to reduce airborne fibre levels and hence the dose, by producing a fibre that has an increased solubility rate in physiological solutions. This is not a simple challenge, as can be seen from table 2, since increasing components in a vitreous fibre that will increase the solubility rate will tend to reduce refractoriness and therefore, the use temperature of any ensuing fibre. The development of such a fibre, Superwool X607, is presented in this paper. As solubility rate in physiological solutions is an important aspect in this development, the in-vitro solubility rate tests that were used during this work at both Manville Mountain Technical Centre (MMTC), Denver, TX (USA), and at Morgan Materials Technology Limited (M^2T), Stourport-on-Severn (UK), will first be outlined.

2. In-vitro testing

Two types of test procedure for assessing the potential for fibre dissolution in physiological solutions were developed at both sites and used during the development phase of Superwool X607[1]. The first, a screening test lasting no more than a day, was used to rank the relative solubility rates of a number of compositions within a particular area of a phase diagram of interest by the amount of "silicate" going into solution during the test. In these 0.5 g of material, made on the small-scale spinning or blowing equipment at MMTC or M^2T, respectively, was added to Gambles solution or Saline 3 solution (table 3) in a plastic bottle/tube. At MMTC [7] this was placed in an ultrasonic bath connected to a timer to cycle the bath on for 15 min each hour for a total of 5 h, so as to give a temperature of 40 °C at the end of the test. At M^2T [8 and 9] the centrifuge tube was placed in a shaker water bath at (37±1) °C (body temperature) for 5 and/or 24 h. In both laboratories, at the conclusion of the test, the solutions were immediately decanted from the fibre and the concentration of silicon ions and occasionally other ions, were determined by Atomic Absorption Spectroscopy (AAS) or by Inductively Coupled Plasma (ICP).

More detailed solubility rate studies were then carried out on the most promising compositions using a flow through solubility rate test, lasting several weeks. These flow through tests allowed dissolution rates to be determined, and to prove that the dissolution of the composition was not a short-term initial phenomenon. The development and refining of these tests is ongoing as evidenced by a recent discussion paper on flow through testing [10].

0.5 to 2 g of deshotted fibre was placed in sample holders within an incubator held at (37 ± 1) °C. Gambles or SBF solution (see table 3), at MMTC [11] or M^2T [12], respectively, was pumped at a constant rate of 5 to 10 ml/h through individual sample holders containing fibre, interacts with the fibre and flows out into collection bottles. Solutions from the collection bottles were analyzed periodically for silicon, and other ions as appropriate, by ICP or AAS. These were used to calculate a rate constant K in ng/(cm² h), mass dissolved per unit surface area per unit time, standardized using Specific Surface Area (SSA) measured on the initial sample.

3. History of X607 development

The initial work that eventually brought about the development of X607 began over 20 years ago at MMTC. As can be seen from the general overview of this development given in table 4, it was not originally intended to produce a fibre that would be soluble in physiological

[1] Trademark for product sold by Thermal Ceramics Europe; also sold under Firemaster Trademark.

Table 3. Compositions of the simulated body fluids used at MMTC and M²T, expressed as weight in grammes added to 1 l of distilled water

compound	content of components (in g/l) in simulated body fluids		
	modified Gambles (MMTC)[2)]	Saline 3 (M²T)	Synthetic Body Fluid (SBF) (M²T)
NaCl	6.400	6.780	5.967
$NaHCO_3$	2.000	2.268	0.353
Na_2HPO_4	0.150	0.171	
$MgCl_2 \cdot 6H_2O$	0.160		
Na_2SO_4	0.080		0.071
$CaCl_2 \cdot 2H_2O$	0.060		
$CH_3COONa \cdot 3H_2O$	1.100		
$NaNO_3$	0.500		
NH_4Cl		0.535	
$Na_3C_6H_5O_7 \cdot 2H_2O$		0.059	
$H_2NH_2CCO_2H$		0.450	
H_2SO_4		0.049	
$(NH_4)_2HPO_4$			0.132
KCl			0.224
tris-buffer			4.000
10% w/w solution of 35% HCl			33.2 ml

[2)] Saturated with 5% CO_2/95% N_2 to pH 7.6.

solutions. That it did so is an example of the importance of ongoing research in a company's prime product areas leading to unexpected benefits.

A range of compositions was gradually produced as fibres by spinning a melt stream from a small electrode melter. As the goal of developing a mineral wool with an American Society for Testing and Materials (ASTM) 2 h fire test rating was progressively achieved, it was realized from screening solubility rate tests (see section 2.) during the early 1980s that some of these compositions had some limited dissolution in physiological solutions. With the growing worries about the hazard of fibrous material highlighted by the asbestos issue, and experimental results with ceramic fibre at the Los Alamos National Laboratories [13] and inhalation experiments at the Institute of Occupational Medicine [14], increased dissolution became an important property of these new fibre compositions. As a consequence, in 1985 the emphasis of the project was changed to that of producing a 2 h fire-rated mineral fibre that was soluble in physiological solutions. The first patent outlining a composition area that satisfied these two goals was filed in the USA in 1986. In 1987 the increase in solubility rate obtained by lowering the Al_2O_3 level was highlighted, along with the first indication of a composition region where the normal effect of Al_2O_3 on refractoriness as given in table 2 did not apply. Reducing the Al_2O_3 level from 20 to 10 wt% apparently increased the use temperature of the fibre. Patent protection was sought at appropriate stages during the development process [15].

During the protocol development of the animal inhalation studies [16 to 19] on aluminosilicate refractory ceramic fibre (RCF), it was considered that it would be sensible to include a different type of fibre. It was therefore decided to test the hypothesis that a fibre with a higher solubility rate in a physiological solution should be less durable and therefore, should show less tendency to induce fibrosis and tumours. A composition, X607-0 (figure 1), was chosen and a maximum tolerated dose study carried out at Research and Consultancy Company (RCC), Geneva (Italy) [20]. The conclusion from the study was that the reaction in the rats' lungs was no more than would have been expected from an inert dust. Compared with the other RCF fibres used in the RCC studies, as has been presented at two workshops [21 and 22], the significant difference in biological outcome may be related to the greater in-vivo dissolution rate of X607.

Two compositions, X607-1 and X607-2 (figure 1), were selected for a production trial at the Manville Waukegan plant in July 1989. They were both successfully and economically spun and evaluated in terms of the properties of the blanket product that was made. The required fire rating was confirmed, and all the other properties needed from a commercially useful blanket product were achieved. This type of product was considered to have a maximum use temperature of 830 °C.

With the decision by Manville to divest itself of its RCF fibre business, the interest in the X607 compositions declined as these were made using the same spinning process lines that were going to be sold. In 1990 the RCF business in Europe and the USA were sold to the Morgan Crucible Company plc. The rights to a fibre that was soluble in physiological solutions, had successfully passed through the RCC rat studies and had a use temperature of 830 °C were also offered. These rights to the X607 technology were acquired in July 1991.

Further development of the X607 family was undertaken at M²T and at Thermal Ceramics de France, St. Marcellin. A large number of compositions were produced on a small-scale rig at M²T by blowing a melt stream, rather than the spinning carried out at MMTC. Taking the concept of maintaining a low Al_2O_3 content to the extreme it was found that, besides giving at least the very good solubility rate of X607-0 in physiological

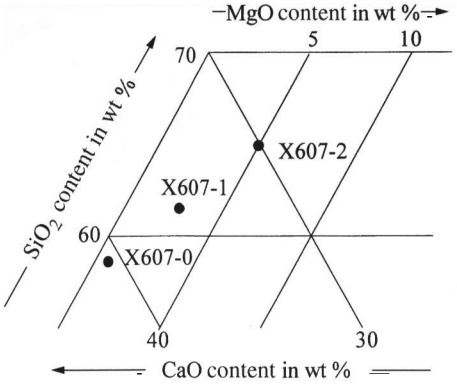

Figure 1. Trial and RCC compositions from CaO–MgO–SiO_2 phase diagram.

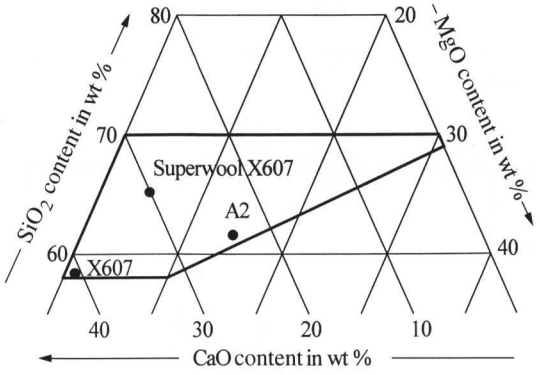

Figure 2. Patented 1000 °C use temperature area of CaO–MgO–SiO_2 phase diagram.

solutions, a large composition area could be defined that had 1000 °C use temperature (figure 2). This use temperature area was defined using a ramp to temperature shrinkage test based on the draft standard ISO/DIS 10 635 [23] proposed by Comité Européen de Normalisation (CEN). UK patent protection for this extension to the X607 philosophy was sought in January 1992, with the finalized version being filed as a PCT application in January 1993 [8].

Table 5. Solubility rate constants comparing Superwool with some of the MMVF materials tested at RCC and some natural mineral fibres

material	solubility rate constant in ng/(cm² h)	
	studies at MMTC (21 d flow through tests)	typical values/ranges from other studies [10, 22, 24 and 25]
erionite	=	0.002
crocidolite	=	0.1 to 0.2
chrysotile	=	0.05
MMVF-21	=	15 to 33
MMVF-11	98	80 to 150
X607-0	190	185
MMVF-10	=	250 to 450
Superwool X607-spun	610	=

Optimization of the processing of these types of fibre in terms of both economics and fibre quality/properties at Thermal Ceramics de France led to the launch of the Superwool™ X607 composition (see figure 2) in 1992. The good solubility rates from flow testing (see section 2.) of this material and X607-0 compared with the glass-wools (MMVF-10 and 11) and rockwool (MMVF-21) tested in RCC studies [18 and 19], and natural mineral fibres (crocidolite, chrysotile and erionite) are shown in table 5.

Two compositions, one near the centre (Superwool X607) and one near the right hand edge (A2) of the 1000 °C use temperature area (figure 2), were used in an intratracheal instillation durability study at the Biomedical Research Laboratories of AEA Technology, Harwell (UK) [26]. After 180 d the number of fibres remaining in the lungs had reduced to ≈10 % of the initial burden for both fibre compositions. The time for the initial burden to reach 50 % of the initial burden, $t_{1/2}$, was between 30 and 40 d. This is comparable with the value obtained on X607-0 in a similar intratracheal instillation study in another laboratory [27 and 28]. This means that similar in-vivo behaviour has been observed for three different compositions of the Superwool family. As can be seen

Table 4. Development of Superwool X607

company	year	subject of development	year of patent filing	main component (in wt%) of fibre composition	
				silicon	aluminium
Manville Mountain Technical Centre	since 1975	mineral wool to pass 2 h fire test		44	7 to 15
	since 1985 to 1989	emphasis changed to solubility benefit	1986 to 1988	52 to 59 to 60	7.8 to 1
Morgan Materials Technology	since 1991	improving refractoriness whilst maintaining solubility	1992	59 to 60	1
	in 1993	launch of Superwool X607	1993	65 to 66	0.5

from figure 2, the composition of the RCC tested fibre, X607-0, is in one corner of the composition area that has 1000 °C use temperature. The results on the two compositions tested at Harwell, one near the centre (Superwool X607) and one near the right hand edge (A2) of the area, when combined with the data from the other intratracheal study on X607-0, provide a strong case for the extrapolation of in-vivo inhalation data obtained on one fibre in this series to any other fibres in the Superwool family. Consequently, the negative results from the inhalation study at RCC on X607-0 can be assumed to equally apply to the A2 composition and to Superwool X607. Interestingly, a recent inhalation recovery experiment [22] on X607-0 has shown an even lower $t_{1/2}$ value than for these intratracheal studies, which reinforces the high solubility of this type of fibres giving rapid in-vivo clearance.

4. Future developments in "soluble" fibres

From the investigations on the Superwool composition families and other potential "soluble" fibre systems, rules have emerged as to how to predict compositions that should form silicate fibres that will be soluble in physiological solutions. These rules have been incorporated in the 1992 UK and 1993 PCT [8] patent applications mentioned in section 3.

The first rule that can be used to predict solubility rate is based on the sum of the free energy of hydration of the individual phases that can be considered to make up any chosen composition. The sum of all the ions that dissolve into a simulated body fluid in a 24 h "static" test, expressed as parts per million (ppm) of oxide (ppm SiO_2 + ppm MgO + ppm CaO +...), gives the total amount of soluble species for each composition. The logarithm of this total amount of soluble species is related to the calculated free energy of hydration (figure 3). The higher the free energy of hydration, the more energetically favourable is the dissolution of the material, and compositions with a free energy more negative than − 400 kJ/kg have always shown good solubility rates.

However, as can be seen from the data in figure 3, the experimental results give a general trend, rather than a good linear fit. A much better fit has been achieved for the second rule by relating the same logarithm of the total solubility rate data (ppm in a 24 h test) to the percent of non-bridging oxygens (NBOs) associated with the particular glass composition (figure 4).

The extent of NBOs gives a measure of the disruption of the three-dimensional silicate network by modifying cations. The creation of NBOs is shown in figure 5 for a modifying calcium ion.

The application of these solubility rate rules, combined with empirical knowledge of glass and fibre forming compositions has led to a number of "soluble" fibre families with the potential for use at relatively high temperatures. Many suffer from viscous flow effects that give

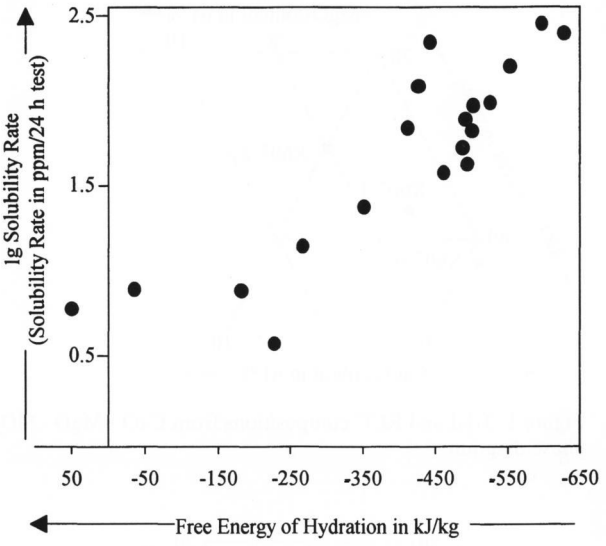

Figure 3. Relationship between solubility rate and free energy of hydration.

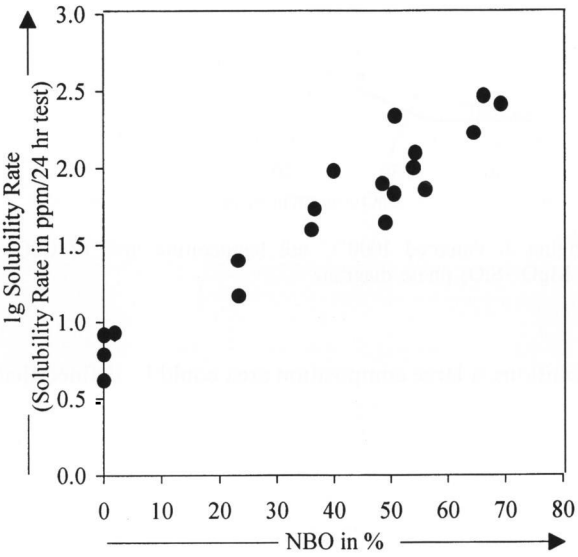

Figure 4. Relationship between solubility rate and percentage of non-bridging oxygens (in % NBO).

excessive shrinkage at temperatures between 800 and 1000 °C, making them no better than a standard mineral wool product. Other systems have use temperatures comparable with Superwool X607, but comprise of more expensive raw materials and have no other practical benefits, and so there has been no reason to commercialize these. However, there still remain several systems that appear to have higher use temperatures than Superwool X607, whilst still maintaining a reasonable solubility rate in physiological solutions. One such system is the subject of a recently published PCT patent application [29]. In the ongoing work to optimize these compositions the problem highlighted in table 2 is always present. As higher refractoriness is aimed for, there is a tendency to reduce the solubility rate of the resultant fibre, often

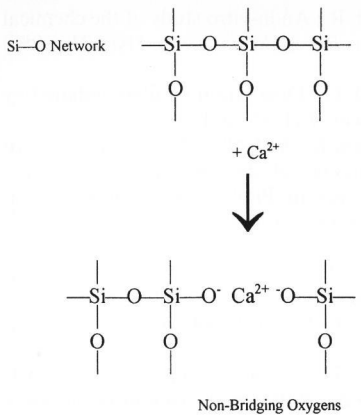

Figure 5. Development of non-bridging oxygens due to incorporation of calcium in a silicate network.

dramatically. Nevertheless, a 1260 °C grade fibre, with a solubility rate significantly greater than current RCF, that can be made into all the currently required product forms, is a possibility that may be achievable as a commercial product in the foreseeable future.

5. Conclusions

From a project that started over 20 years ago, a new fibre product, Superwool X607, is now available in all the normal forms of blanket, board, shapes, rope and paper. Superwool X607 is soluble in physiological solutions, a related composition has passed a maximum tolerated dose rat inhalation study, and has a use temperature of 1050 °C.

The hypothesis that a fibre that is soluble in physiological solutions will be less durable in the lungs, and therefore has a lower propensity to cause fibrosis and tumours, even at excessive dose levels, has been validated for one relatively high solubility rate material. The concept that fibres from a compositional family that have similar solubility rates in physiological solutions will have comparable in-vivo durabilities has been shown to be valid from intratracheal instillation studies for one relatively high solubility rate composition family.

The understanding that has been developed from the work on the Superwool X607 family, has helped in the engineering of another fibre family which may have a use temperature of up to 1260 °C, whilst maintaining a reasonable solubility rate in physiological solutions. The refining and optimizing of this fibre family into a commercial product has still to be achieved.

High-temperature "soluble" fibres are a reality that are here and the range of them will grow to fill the perceived need in the market.

*

The permission of the Mountain Technical Centre of Schuller International Inc., Denver, CO (USA), (formerly MMTC) to include information from their development work on X607 and the unpublished RCC report on the experiments on X607 is gratefully acknowledged.

6. References

[1] International Agency for Research on Cancer (IARC): Man made mineral fibres and radon. IARC monographs on the evaluation of carcinogenic risks to humans. Vol. 43. Lyon: IARC, 1988.

[2] National Occupational Health & Safety Commission, Worksafe Australia. Technical report on synthetic mineral fibres. In: Technical report on synthetic mineral fibres & guidance note on the membrane filter method for the estimation of airborne synthetic mineral fibres. WAP 89/013. Canberra: Australian Government Publ. Service, 1989. p. 1–80.

[3] Kotin, P.: A historical perspective of fiber-related disease. In: Warheit, D. B. (ed.): Fiber toxicology. San Diego (et al.): Academic Press, 1993. p. 1–14.

[4] Pott, F.: Testing the carcinogenicity of fibers in laboratory animals: results and conclusions. In: Warheit, D. B. (ed.): Fiber toxicology. San Diego (et al.): Academic Press, 1993. p. 395–424.

[5] Christensen, V. R.; Jensen, S. L.; Guldberg, M. et al.: Effect of chemical composition of man-made vitreous fibers on the rate of dissolution in vitro at different pHs. Environ. Health Perspect. **102** (1994) suppl. 5, p. 83–86.

[6] Association des Producteurs de Fibres de Verre Européens / European Insulation Manufacturers Association / European Ceramic Fibres Industry Association (APFE/EURIMA/ECFIA): Man-made mineral fibres: definitions and classification proposals. Brussels: APFE/EURIMA; Paris: ECFIA, 1991, p. 15.

[7] Olds, L. E.; Kielmeyer, W. H.: Inorganic fiber composition. Canad. pat. no. 1271785. 1990.

[8] Jubb, G. A.; Martin J.-L.: Saline soluble inorganic fibres. Patent Cooperation Treaty (PCT) appl. no. PCT/GB93/00085. Int. publ. no. WO 93/15028. 1993.

[9] Hoskins, J. A.; Brown, R. C.; Alexander, I. C.: Biological persistence of mineral fibres found as contaminants of indoor air. Environ. Technol. **13** (1992) p. 361–366.

[10] Guldberg, M.; Christensen, V. R.; Krøis, W. et al.: Method for determining in-vitro dissolution rates of man-made vitreous fibres. Glastech. Ber. Glass Sci. Technol. **68** (1995) no. 6, p. 181–187.

[11] Law, B. D.; Bunn, W. B.; Hesterberg, T. W.: Solubility of polymeric organic fibers and manmade vitreous fibers in Gambles solution. Inhal. Toxic. **2** (1990) p. 321–339.

[12] Alexander, I. C.; Brown, R. C.; Jubb, G. A. et al.: Durability of ceramic and novel man-made mineral fibers. Environ. Health Perspect. **102** (1994) suppl. 5, p. 67–71.

[13] Smith, D. M.; Ortiz, L. W.; Archuleta, R. F.: Long-term exposure of Syrian hamsters and Osborne-Medel rats to aerosolized 0.45 µm mean diameter fibrous glass. In: Biological effects of man-made mineral fibres. Proc. WHO/IARC Conference, Copenhagen 1982. Vol. 2. Copenhagen: WHO, 1984. p. 253–272.

[14] Davis, J. M. G.; Addison, J.; Bolton, R. E. et al.: The pathogenic effects of fibrous ceramic aluminium silicate glass administered to rats by inhalation or peritoneal injection. In: Biological effects of man-made mineral fibres. Proc. WHO/IARC Conference, Copenhagen 1982. Vol. 2. Copenhagen: WHO, 1984. p. 303–322.

[15] Olds, L. E.; Kielmeyer, W. H.: Inorganic fiber composition. US patent no. 5 332 699. 1994.

[16] Glass, L. R.; Mast, R. W.; Hesterberg, T. W. et al.: Sub acute (28 day) nose only inhalation study of size selected refractory ceramic fibre (RCF) in male Fischer 344 rats. Toxicologist **13** (1993) p. 34.

[17] Glass, L. R.; Brown, R. C.; Hoskins, J. A.: Health effects of refractory ceramic fibres: scientific issues and policy considerations. Occup. Environ. Med. **52** (1994) p. 433–440.
[18] Bunn, W. B.; Bender, J. R.; Hesterberg, T. W. et al.: Recent studies of man-made vitreous fibers. Occup. Med. **35** (1993) p. 101–113.
[19] Hesterberg, T. W.; Miller, W. C.; McConnell, E. E. et al.: Chronic inhalation toxicity of size-separated glass fibers in Fischer 344 rats. Fund. Appl. Tox. **20** (1993) p. 464–476.
[20] Alexander, I. C.: In vivo studies related to the development of Superwool X607, a soluble high temperature insulation fibre. Occup. Environ. Med. (In prep.)
[21] McClellan, R. O.; Miller, F. J.; Hesterberg, T, W. et al.: Approaches to evaluating the toxicity and carcinogenicity of man-made fibers. Summary of Workshop on Toxicity & Carcinogenicity of Man-Made Fibers, Durham, NC 1991. Regul. Toxicol. Pharmacol. **16** (1992) p. 321–364.
[22] Bernstein, D. M.; Morscheidt, C.; Tiesler, H. et al.: Evaluation of the biopersistance of commercial and experimental fibers following inhalation. Inhalation Toxicol. **7** (1995) p. 1031–1058.
[23] International standard ISO/DIS 10635 (April 1997): Refractory products; method of test for ceramic fibre products. Geneva: ISO, 1997.
[24] Scholze, H.; Conradt, R.: An in-vitro study of the chemical durability of siliceous fibres. Ann. Occup. Hyg. **31** (1987) no. 4B, p. 683–692.
[25] Eastes, W.; Hadley, J. G.: Dissolution of fibers inhaled by rats. Inhalation Toxicol. **7** (1995) p. 179–196.
[26] Morris, K. J.; Launder, K. A.; Collier, C. G. et al.: In vivo and in vitro comparisons of the dissolution of calcium magnesium silicate fibres. In: Proc. 5th International Inhalation Symposium, Hannover 1995. Exp. Toxicol. Pathol. (In prep.)
[27] Muhle, H.; Bellman, B.; Pott, F.: Comparative investigations of the biodurability of mineral fibers in the rat lung. Environ. Health Perspect. **102** (1994) suppl. 5, p. 163–168.
[28] Bellman, B.; Muhle, H.: Biobeständigkeit verschiedener Mineralfasertypen in der Rattenlunge nach intratrachealer Applikation. In: Schriftenr. Bundesanstalt für Arbeitsschutz, Forschung-Fb 711. Dortmund 1995.
[29] Jubb, G. A.: Saline soluble inorganic fibres. Patent Cooperation Treaty (PCT) appl. no. PCT/GB94/00053. Int. publ. no. WO 94/15883. 1994.

1297P004

Address of the authors:

I. C. Alexander, G. A. Jubb
Morgan Materials Technology Ltd.
Bewdley Road
Stourport-on-Severn, Worcestershire DY13 8QR (UK)