Comment on "The crystal structure of the silicon cerium oxynitride $Ce_4Si_2O_7N_2$"

The alternative indexing proposed by Guha [1] for the $Ce_4Si_2O_7N_2$ phase reported by Mah *et al.* [2] is in good agreement with our data for this phase (see Table I, after Buang [3]) obtained during a detailed study of phase relationships in the Ce–Si–O–N system. The unit cell proposed by Mah *et al.* (subscript M) is related to the correct cell (Guha, subscript G) by:

$$a_G = 2c_M, \quad b_G = b_M, \quad c_G = a_M + c_M$$

(see Fig. 1)

and differs only in the periodicity along the a_G axis. The structure of $Ce_4Si_2O_7N_2$, which is similar to $Ca_4Si_2O_7F_2$ [4] and $Eu_4Al_2O_9$ [5], shows strong pseudo-halving in this direction which results in very weak intensities for hkl reflections with h odd. Most of the X-ray pattern can, therefore, be indexed using an a repeat distance of half the true value. Because the β angle in the Mah unit cell is very nearly 90°, there is an alternative monoclinic unit cell with parameters: $a = 7.952$ Å, $b = 10.876$ Å, $c = 11.118$ Å; $\beta = 111.26°$, which is equally correct and has been used by Buang [3].

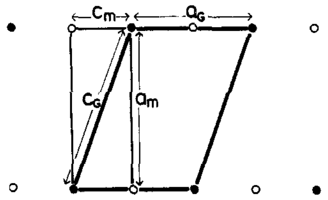

Figure 1 The (0 1 0) plane of $Ce_4Si_2O_7N_2$. There is a simple geometrical relationship between the correct unit cell (subscript G) and the cell proposed by Mah *et al.* [2] (subscript M).

Marchand *et al.* [6] speculate on whether the nitrogen in $Ln_4Si_2O_7N_2$-type phases occupies the fluorine atom sites in $Ca_4Si_2O_7F_2$ or whether it is randomly distributed among SiO_4 tetrahedra. Lang *et al.* [7] have shown that nitrogen apatites of the type $M_{10}(SiO_4)_6N_2$ (M = Ln, Y) are more correctly written $M_{10}(Si_6O_{22}N_2)O_2$ since oxygen preferentially occupies the three-planar co-ordinated anion site. In the cuspidine structure [4], fluorine atoms occupy four-fold sites co-ordinated only by calcium, whereas oxygen atoms occupy four- and three-fold sites co-ordinated by both calcium and silicon. In the analogous europium aluminate structure [5], both types of non-metal sites are occupied by oxygen. In

TABLE I X-ray diffraction data for $Ce_4Si_2O_7N_2$ Monoclinic: $a = 7.952$ Å, $b = 10.876$ Å, $c = 11.079$ Å; $\beta = 110.73°$

hkl	d_{calc}	d_{obs}	I_{obs}
$0\,1\,1$	7.488	7.490	m
$0\,1\,\bar{1}$	6.112	6.113	w
$0\,2\,0$	5.432	5.432	mw
$1\,0\,\bar{2}$	5.202	5.192	w
$0\,0\,2$	5.181		
$0\,2\,1$	4.815	4.813	w
$0\,1\,2$	4.677	4.677	s
$0\,2\,2$	3.744	3.744	mw
$2\,1\,\bar{1}$	3.735		
$2\,0\,0$	3.719	3.720	w
$2\,1\,0$	3.519	3.516	w
$2\,1\,\bar{2}$	3.508		
$0\,3\,1$	3.422	3.416	mw
$0\,1\,3$	3.292	3.294	m
$2\,2\,\bar{1}$	3.210	3.208	vs
$2\,2\,0$	3.070	3.069	w
$2\,2\,\bar{2}$	3.062	3.063	w
$2\,1\,1$	3.037	3.035	w
$2\,1\,\bar{3}$	3.022	3.022	w
$0\,3\,2$	2.970	2.971	m
$0\,2\,3$	2.916	2.915	s
$2\,2\,\bar{3}$	2.723	2.720	w
$0\,4\,0$	2.719		
$0\,4\,1$	2.629	2.630	mw
$2\,0\,2$	2.614	2.614	mw
$2\,0\,\bar{4}$	2.601	2.600	mw
$2\,3\,0$	2.596	2.596	w
$2\,3\,\bar{2}$	2.591		
$0\,0\,4$	2.590		
$2\,1\,2$	2.542	2.542	w
$2\,1\,\bar{4}$	2.529	2.529	w
$0\,1\,4$	2.520	2.521	vw
$2\,3\,1$	2.383	2.380	w
$2\,3\,\bar{3}$	2.376		
$2\,4\,\bar{1}$	2.244	2.245	mw
$0\,5\,1$	2.129	2.129	vw
$2\,3\,2$	2.121	2.121	mw
$2\,3\,\bar{4}$	2.113	2.114	mw
$2\,4\,1$	2.062	2.061	mw
$2\,4\,\bar{3}$	2.057		
$0\,1\,5$	2.036	2.036	m
$4\,0\,\bar{2}$	1.988	1.988	m
$0\,2\,5$	1.937	1.939	w

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$Ce_4Si_2O_7N_2$ it seems more likely that nitrogen is distributed statistically among the non-metal sites rather than preferentially occupying the cerium co-ordinated sites, but a complete structure determination would be necessary to confirm this.

A more detailed discussion of the Ce–Si–O–N system will be published separately.

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Fatigue properties of high density polyethylene pipe systems

Plastics pipe-networks are composed of extruded pipe and injection-moulded fittings, and network lifetime assessment concentrates on testing the pipe only, under conditions of static fatigue (i.e. creep). Early work [1] on a high-density polyethylene (HDPE) pipe showed that dynamic, as opposed to static, fatigue did not reduce pipe lifetime, where lifetime is defined by the time under maximum load. However, recent work [2] extended testing to HDPE pipe systems under conditions of dynamic square wave fatigue at a fixed frequency of 4 cycles per minute (cpm) that is $7\frac{1}{2}$ sec on, $7\frac{1}{2}$ sec off; at this fixed frequency pipe system lifetime was significantly reduced in comparison to the creep life of the pipe, and failure was shown to be due, in part, to the incorporation of injection-moulded fittings.

An extensive and systematic study of the performance of a range of mirror-plate butt-welded HDPE pipe systems under burst, creep and fatigue loadings has been undertaken at Brunel University with the aim of identifying more precisely, the lifetime of pipe-networks subject to these various loading profiles. Work centred initially on a HDPE resin, of pipe density 0.954 g cm^{-3}, fabricated into a 63 mm SDR 11 system, incorporating extruded pipe and injection-moulded flanges, 90° bends and 90° equal tees. This diameter of system was selected for reasons of economy and ease of handling. The pipe systems were assembled to dimensions to conform to the minimum requirements of ASTM standards D2610 and D1598 by mirror-plate butt-welding under optimum conditions of weld temperature and pressure. The details of the test procedures and the geometry and fabrication techniques of the pipe systems will be presented in future publications [3]. So too will be further creep and fatigue data and fractographic studies on this 63 mm system, together with data on other pipe systems. This note serves only to demonstrate that pipe system lifetime can be strongly frequency-dependent over the limited frequency range (up to 10 cpm) of the fatigue loadings studied; in this respect the present work supports the observations of Cowley and Wylde [2], that pipe-network lifetime can depend on the integrity of the injection-moulded fittings or welds between fittings and pipe, as opposed to the strength of the extruded pipe.

Schematic diagrams of the principal 63 mm pipe systems tested are illustrated in Fig. 1. This note reports the results of those tests where the systems were maintained at a temperature of 80° C (353 K), by immersion in a constant temperature (± 1.0 K) water bath, and pressurized to 1.00 MPa