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ANALYTICAL ELECTRON MICROSCOPY OF SHOCK-SYNTHESIZED NIOBIUM SILICIDE COMPOSITES

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In-situ composites of thermodynamically compatible phases have been the subject of recent investigations[1] and in particular, those comprising 2-phase Nb/Nb_5Si_3 structures.[2] Chemical reactions initiated by high velocity shock waves can be extremely rapid and (unlike more conventional techniques) are capable of forming composite compounds with carefully controlled, fine-scale microstructures. The current investigation involves the use of SEM, TEM and EDX to fully characterize the microstructure of niobium silicide specimens (of initial bulk compositions Si 40 at.% Nb and Nb 33 at.% Si) formed by shock-synthesis of nominally 5 μm size particles in a powder gas gun at an impact velocity of 1.3 km/s.

Recovered specimens were in the form of 34 mm diameter × 7 mm thick disks. Metallographic sectioning and examination by SEM in the backscattered imaging mode showed that both disks had undergone a chemical reaction with the exception of the peripheral regions which were merely compacted. Examination was also performed using a Philips CM30 analytical TEM at 300 keV in addition to EDX with a Link Systems ultrathin window detector. Spectra were quantified using the ratio technique[3] and with experimental k-factors.

Spheroidal precipitates of $NbSi_2$ formed at the Si/Nb interfaces in both specimens and were indicative of the first stages of a chemical reaction (shown by the intermediate contrast in Fig.1 and by the EDX spectrum in Fig. 3). The microstructure in the central region of the 60 at.% Si specimen consisted of a $NbSi_2$ matrix containing Nb-metal particles which were each surrounded by a shell of Nb_5Si_3 (as shown in Fig. 2). By contrast, the microstructure in the central region of the 67 at.% Nb specimen consisted of small residual Nb particles in a matrix of Nb_5Si_3 together with a eutectoid type reaction product of the type shown in Fig. 4. Chemical analyses of the two components of the fine-scale eutectoid structure were performed on a thin foil specimen (Fig. 5). Unlike the SEM, the fine probe size and small interaction volume in TEM permitted the unambiguous identification of the two constituent phases as Nb and Nb_5Si_3 (as shown by the spectrum in Fig. 6). The k-factors and chemical compostions of the two silicide phases are shown in Figs. 3 and 6.

The current investigation has shown, through the use of AEM in the characterization of Nb-silicides, that if shock synthesis is to be used to fabricate such materials, care should be exercised in the choice of the mean initial composition (to retain a 2-phase structure) and that the particle size of the tough metallic phase should be sufficiently large to prevent the formation of an equilibrium eutectoid structure.[4]

References

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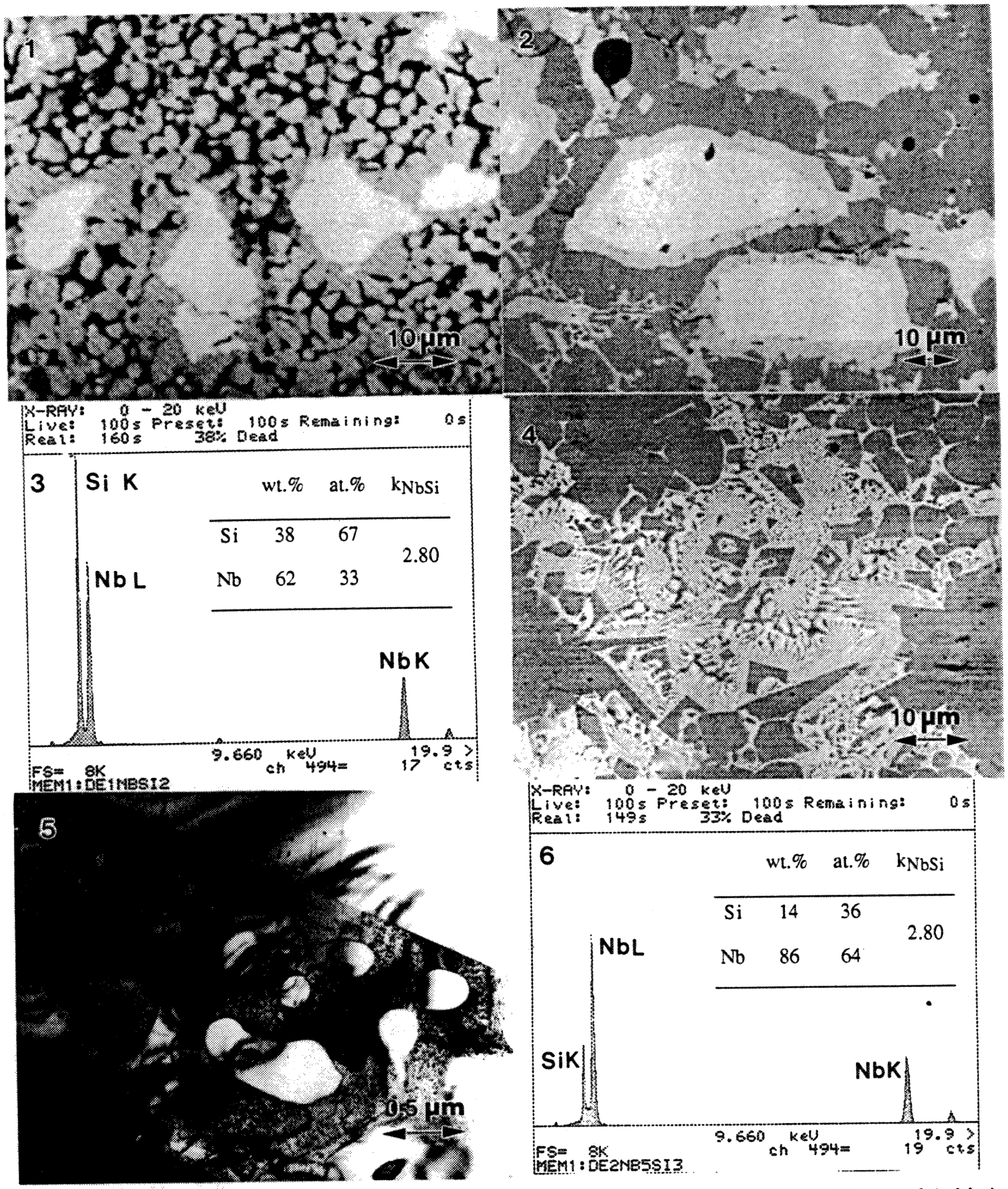

FIG. 1 – Backscattered SEM image showing spheroidal particles of $NbSi_2$ (grey) and Nb-metal (white).
FIG. 2 – SEM image from center of 60 at.% Si specimen showing white Nb particles, 'shell'-type zones of Nb_5Si_3 in a matrix of $NbSi_2$.
FIG. 3 – EDX spectrum from $NbSi_2$.
FIG. 4 – Backscattered SEM image of eutectoid structure in center of 67 at.% Nb specimen.
FIG. 5 – Bright field TEM image of structure shown in FIG. 4.
FIG. 6 – EDX spectrum from Nb_5Si_3 phase - shown by lighter regions in FIG. 5.