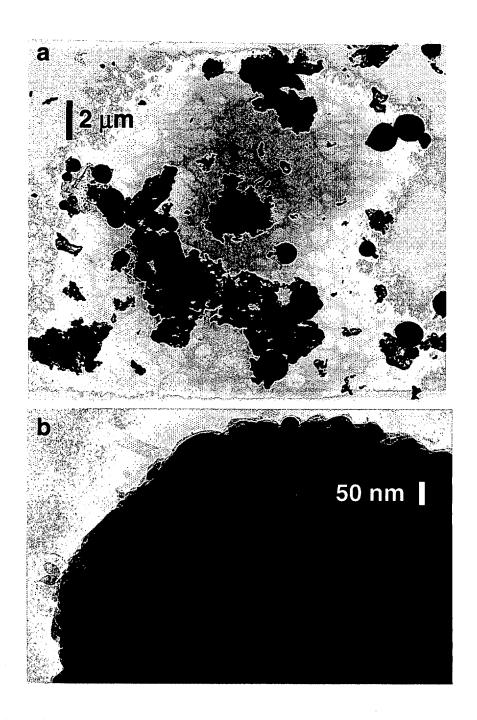


**Figure S1.** TEM image of MoSi<sub>2</sub> obtained from eq 5 in the absence of diluent, which reveals particle sizes in the 100-200 nm range and a degree of interparticle sintering.



**Figure S2.** TEM images that reveal the agglomerate structure of a powder prepared with a LiCl diluent (LiCl:MoCl<sub>3</sub> ratio = 2.5) and annealed for 12 h at 900 °C. The XRD pattern in Figure 2h and the images in Figure 3 were collected from the sample used here: (a) a combination of dense, spherical agglomerates and less dense, irregular agglomerates, (b) a close-up of a spherical aggregate.

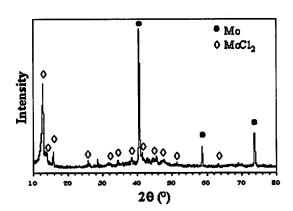
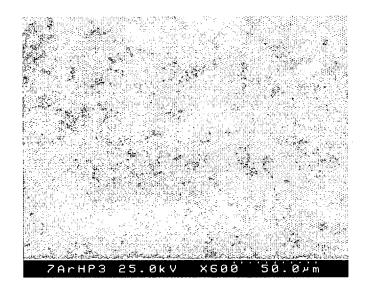


Figure S3. An XRD pattern of a powder obtained from eq 6. The average grain size for Mo is 73 nm. EDS analysis indicated that this mixture contained approximately 10 atom % Si.



**Figure S4.** An SEM image of the polished, unetched surface of a 95%-dense MoSi<sub>2</sub> compact that reveals a very low level of fine porosity. The image in Figure 6 was collected from the same specimen, after etching.