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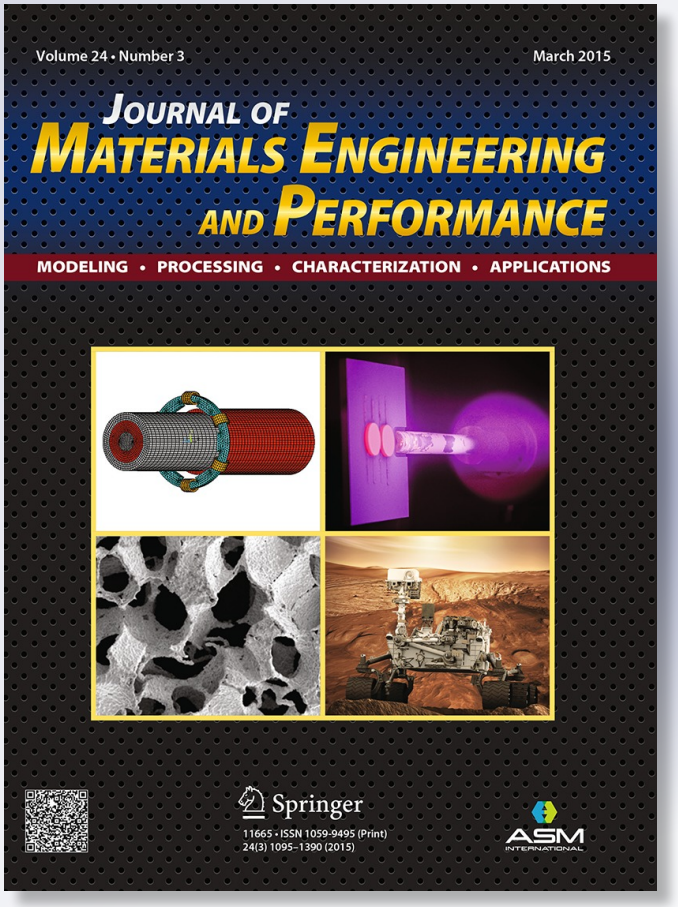
Journal of Materials Engineering and Performance

ISSN 1059-9495
Volume 24
Number 3

J. of Materi Eng and Perform (2015)
24:1199-1205
DOI 10.1007/s11665-015-1390-8



Springer

JMEPEG (2015) 24:1199–1205
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1059-9495/$19.00

Comparison of the Thermal Expansion Behavior of Several Intermetallic Silicide Alloys Between 293 and 1523 K

S.V. Raj

(Submitted November 12, 2014; in revised form December 19, 2014; published online January 21, 2015)

Thermal expansion measurements were conducted on hot-pressed $CrSi_2$, $TiSi_2$, WSi_2 and a two-phase Cr-Mo-Si intermetallic alloy between 303 and 1523 K during three heat-cool cycles. The corrected thermal expansion, $(\Delta L/L_0)_{thermal}$, varied with the absolute temperature, T, as

$$(\Delta L/L_0)_{thermal} = A(T-293)^3 + B(T-293)^2 + C(T-293) + D$$

where, A, B, C, and D are regression constants. Excellent reproducibility was observed for most of the materials after the first heat-up cycle. In some cases, the data from first heat-up cycle deviated from those determined in the subsequent cycles. This deviation was attributed to the presence of residual stresses developed during processing, which are relieved after the first heat-up cycle.

Keywords CTE, disilicides, intermetallic alloys, thermal expansion

1. Introduction

Intermetallic silicides have found applications as ohmic contacts in the semiconductor industry (Ref 1–5), thermoelectric materials (Ref 6), heating elements (Ref 7, 8), protective coatings (Ref 9), and they have been proposed for structural applications (Ref 10). A knowledge of the thermal expansion behavior of these silicides is important in these applications in order to reduce or eliminate thermal stresses between the intermetallic silicide and the substrate. Although a compilation of the thermal expansion data on many intermetallic silicides is available (Ref 11), the data are fairly old and it is unclear whether they were influenced by impurities and processing methods. Verkhorobin and Matyushenko (Ref 12) reported limited data on the coefficients of thermal expansion (CTE) of $CrSi_2$, $MoSi_2$, $NbSi_2$, $TaSi_2$, and VSi_2 determined from x-ray data. A more complete thermal expansion data on transition metal silicides also determined by x-ray diffraction (XRD) have been reported by Engström and Lönnberg (Ref 13). In contrast, there are only limited thermal expansion data on hot-pressed silicides generated by dilatometric measurements.

The present investigation was undertaken to determine and compare the thermal expansion behavior of hot-pressed $CrSi_2$, $TiSi_2$, WSi_2, and a two-phase Cr-30(at.%)Mo-30%Si* intermetallic alloy between 303 and 1523 K by dilatometric measurements. It is noted that there is no previous CTE data for the Cr-30Mo-30%Si alloy.

2. Experimental Procedures

Commercially produced powders (−325 mesh) of $CrSi_2$, $TiSi_2$, WSi_2, and a Cr-30%Mo-30%Si alloy were hot-pressed into 25.4 mm long and 9.5 mm in diameter cylindrical specimens. The Cr-30%Mo-30%Si alloy was procured from ATI Powder Metals, Pittsburgh, PA as gas atomized powder. Table 1 gives the total purity and major impurity content of the powders. The powders were hot-pressed using conditions given in Table 2. Three $CrSi_2$ specimens were fabricated in separate hot-press runs (582, 619, and 620) in order to evaluate the effect of batch-to-batch variabilities on the thermal expansion data. The two faces of hot-pressed specimens were machined to ensure that they were flat and parallel. The thermal expansion measurements were conducted using a NETZSCH Dilatometer Model DIL 402C equipped with a high purity alumina as a calibration standard. Measurements were made over three heat-cool cycles to (a) minimize the effects of compositional, microstructural and processing variables on the data, (b) determine the extent of scatter in the data, and (c) to evaluate a statistical average of the coefficients for the regression curve. The specimen was placed in a sample holder and aligned with a single push-rod with an applied constant load of 0.2 N. The specimens were heated from 303 to 1523 K at 10 K/min in the first cycle and cooled to 373 K at 10 K/min. in the first cool-down cycle. Subsequent cycles consisted of heating and cooling between 373 and 1523 K at 10 K/min. All measurements were conducted in a He atmosphere

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*Unless specifically stated, all compositions are reported in at.% in this paper.

Table 1 Chemical compositions of the silicide powders in wt%

Powder	Al	Cr	Fe	Mo	Ni	Si	Ti	W	Total purity
$CrSi_2$ (Alfa Aesar)	0.05	46.5	0.38	…	0.13	52.4	0.33	…	98.9
Cr-Mo-Si (ATI)	…	35.2	0.038	50	0.015	14.7	…	0.06	99.9
$TiSi_2$ (Cerac)	…	…	…	…	…	…	…	…	99.5
WSi_2 (Materion)	0.005	0.001	0.005	…	…	22.9	…	76.6	99.5

Table 2 Processing data for hot-pressing the silicide powders

Silicide powder	Run #	T, K	P, MPa	Time, h	Environment
$CrSi_2$	582	1183	68.9	2.0	Ar
$CrSi_2$	619	1523	89.6	0.25	Ar
$CrSi_2$	620	1523	89.6	2.0	Ar
Cr-Mo-Si	621	1723	89.6	4.0	Ar
$TiSi_2$	641	1523	89.6	0.67	Ar
WSi_2	622	1573	89.6	2.0	Ar

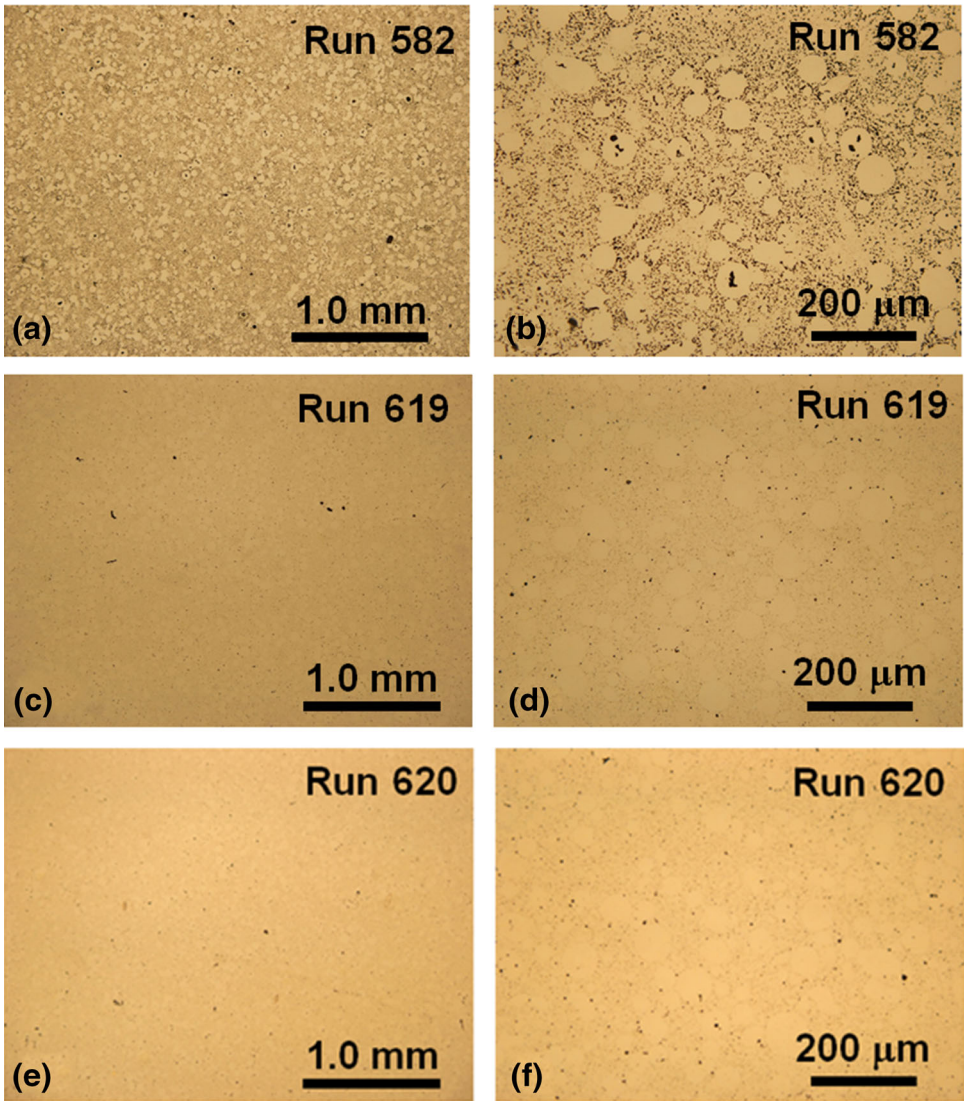

Fig. 1 (a-f) Low and high magnification optical micrographs of the transverse cross-sections of hot-pressed $CrSi_2$ specimens fabricated in three runs

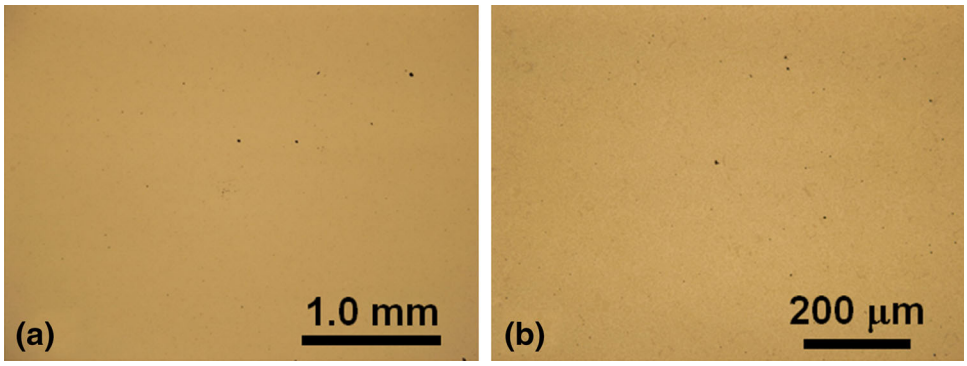

Fig. 2 (a-b) Low and high magnification optical micrographs of the transverse cross-sections of a hot-pressed Cr-30%Mo-30%Si specimen

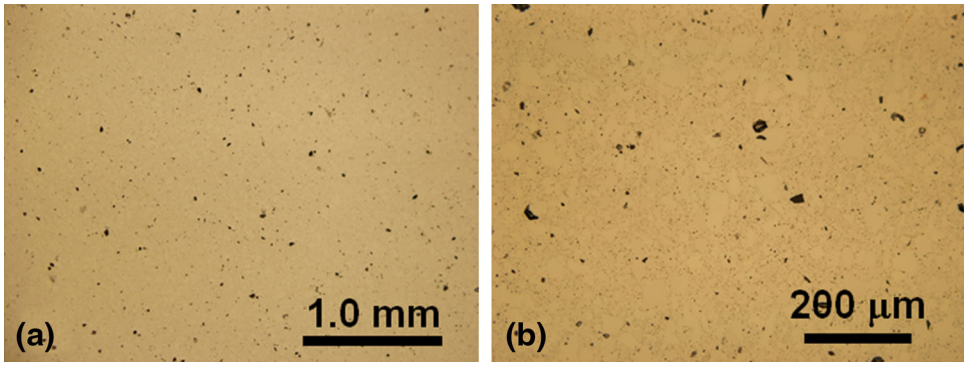

Fig. 3 (a-b) Low and high magnification optical micrographs of the transverse cross-sections of a hot-pressed $TiSi_2$ specimen

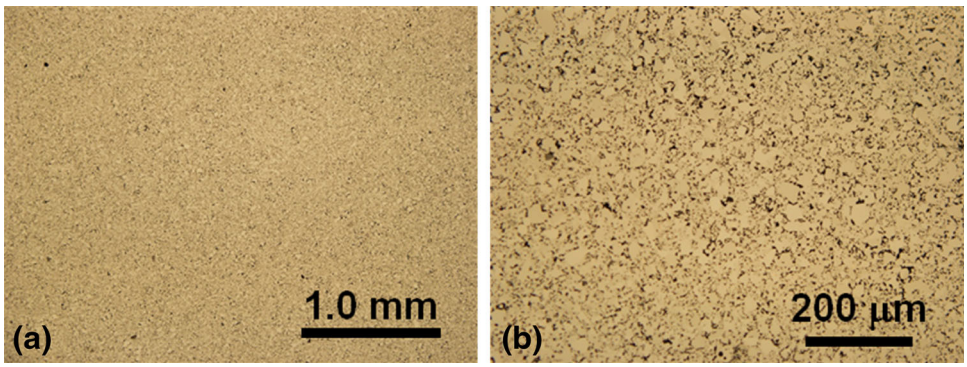

Fig. 4 (a-b) Low and high magnification optical micrographs of the transverse cross-sections of a hot-pressed WSi_2 specimen

flowing at 60 cm^3/min. The length changes were recorded by a computerized data acquisition system. The experimental strain, $\Delta L/L_0$, where ΔL is the differential change in length, $L - L_0$, L is the instantaneous length, and L_0 is the original length of the specimen at room temperature, were measured.

3. Results and Discussion

3.1 Microstructures

Microstructural observations of the hot-pressed specimens revealed that most of them were well consolidated although the extent of homogeneity varied from specimen to specimen. Figure 1(a-f) show optical micrographs of the transverse cross-sections of three hot-pressed batches of $CrSi_2$. While the microstructures for specimens from hot-pressing runs 619 and 620 show a great degree of homogeneity and a distribution of fine grain boundary porosity, the microstructure for run 582 shows the boundaries of the powder particles and extensive porosity. It is noted that run 582 was hot pressed at 1183 K while runs 619 and 620 were hot pressed at 1523 K. The optical microstructures of the hot-pressed Cr-30Mo-30Si alloy were homogeneous and fully consolidated with little porosity (Fig. 2a-b). In contrast, the hot-pressed microstructures of the $TiSi_2$ (Fig. 3a-b) and WSi_2 (Fig. 4a-b) showed a greater amount of grain boundary porosity.

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