MOLYBDENUM DISILICIDE OBTAINING

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Abstract: Molybden disilicide is one of the most known metallic silicides and of its specific properties it can be used in the top of fields of the world-wide techniques. The work deals with the synthesis of molybdenum disilicides. Some information about the evolution of molybdenum disilicide synthesis in varying temperature conditions and the influence of reaction environment are given; the synthesis has been carried out in the random presence of other forms of molybdenum silicides. Other molybdenum silicides which can appear in the process have been shown, too. Concomitantly, the conditions of coexistence for these products are presented. The results are confirmed by the X-ray diffraction analysis and microstructural observations.

Keywords: *molybdenum disilicide, composite material, protective medium, synthesis.*

1. INTRODUCTION

Silicides of transitional metals together with berillides and aluminides belong to the intermetallic compound class.

Some of these silicides have heat-resisting, catalytic and low heat-resistivity properties; for example molybdenum disilicide ($MoSi_2$) which is one of the most known metallic silicides and, because of its specific properties it can be used in the top fields of the world-wide techniques.

The bibliographic references present a lot of methods regarding molybdenum disilicide synthesis depending on various influence parameters[1-12].

The study shows some of our determinations regarding $MoSi_2$ synthesis that have resulted after the research work considering the temperature and protecting environment as influence factors.

2. EXPERIMENTAL WORK

As reactant elements, molybdenum (Mo = 99,25 %, Si = 0,021 %, C = 0,20 %) as very fine powder (below 80 μm) and freshly milled (below 80 μm) silicon (Si = 95,28 %, Fe = 1,82 %, Al = 1,30 %, Ca = 1,01 %) were used.

Table 1 Raw materials used in molybdenum disilicide synthesis

Reactant Elements	Chemical Composition, %										Grinding Fineness
	Mo	Si	S	Fe	C	Mg	Ca	Mn	Cu	Al	
Molybdenum powder	98,3	traces	0,01	0,12	0,26	traces	-	<0,03	<0,01	<0,03	< 30 μ
Silicon	-	96,36	-	1,51	-	traces	traces	-	-	1,28	< 70 μ

The main properties of the reactant elements are shown in Table 1.

The reactant mixture, that had been dosed approximately to molybdenum disilicide stoichiometric formula was heated (like pressed bricks) at various temperatures in the range of 1100-1600^{0}C, under high purity argon and nitrogen protection medium (at 0,3 atm pressure); reaction time at elevated temperature being 20 minutes.

Specific physical-chemical properties were determined on the syntheses obtained in this manner. Phase determination was performed by X-ray diffraction (SEIFERT Cu cathode) analysis and microstructural observation was conducted using a transmission electronic microscope (JEM-100CX).

The quantitative estimation of the synthesized products was made taking into account the intensities (expressed in number of impulses per second) of the main line (d = 0,2020 nm for $MoSi_2$-U, d = 0,2169 nm for $MoSi_2$-H) of studied phases, that correspond to each sample.

3. RESULTS AND DISCUSSIONS

3. 1. Temperature Influence

The X - ray analysis of $MoSi_2$ developing process in argon protective medium, depending on the temperature value showed that at 1150^{0}C molybdenum silicides are not formed. Only Silicon and

Molybdenum are present at this temperature, Figure 1a. and Figure 2.

The molybdenum silicides forming process begins at 1200^{0}C (Figure 1b.), when Molybdenum and Silicon are present, too.

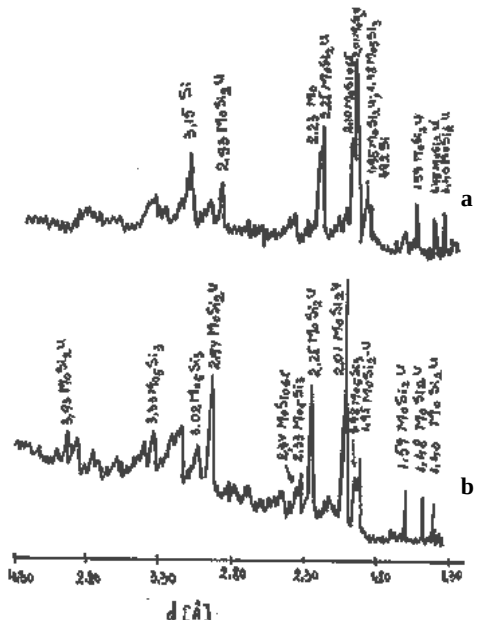

Figure 1. Diffraction analysis of the heated sample (a = 1150^{0}C, b = 1200^{0}C)

The average intensity $MoSi_2$ forming is noticed at 1300^{0}C, and at the same time the presence of the two reactants is not seen any more. At that temperature $MoSi_2$ forms together with Mo_5Si_3 and $MoSi_{0.65}$ (as well as in the 1200^{0}C heating case), only the last phases are observed in a smaller quantity, Figure 3.

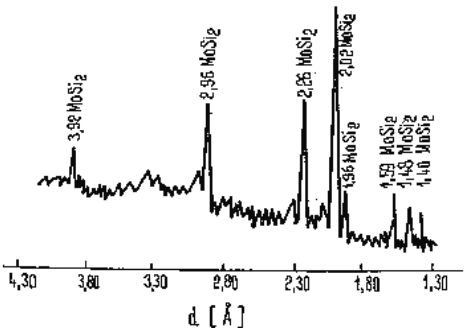

Figure 2. Diffraction analysis of the heated sample at 1500^{0}C

Heat treating the reactant mixture at 1350^{0}C leads to intense $MoSi_2$ forming. At that temperature, Mo_5Si_3 and $MoSi_{0.65}$ continue also to produce, having a strong and average intensity rate.

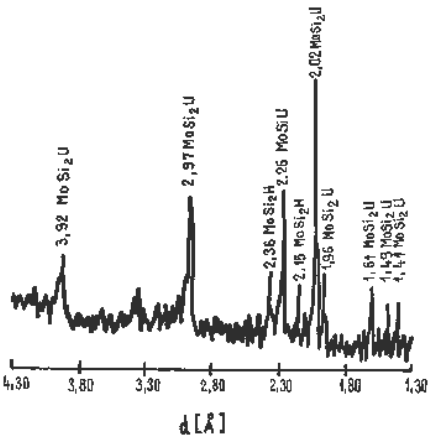

Figure 3. Diffraction analysis of the heated sample at 1600^{0}C.

At 1400^{0}C and 1450^{0}C, $MoSi_2$ is produced with very high intensity together with the intense forming of Mo_5Si_3; the presence of $MoSi_{0.65}$ is not noticed any more.

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The porous structures are involved in these stages1, and strong textural-structural modifications take place, too.

The increase of the reactant mixture temperature up to 1500^{0}C determines only $MoSi_2$ to be produced of very high intensity. Other types of molybdenum silicides have not been noticed at the roentgenographic analysis, Fig. 1c.

4. CONCLUSIONS

The scope of this study is to obtain molybdenum disilicides ($MoSi_2$) by synthesis.

That is why the work shows some of our determinations regarding $MoSi_2$ synthesis that have resulted after the research work considering the temperature and protecting environment as influence factors.

The reactant elements used for the synthesis are molybdenum (Mo = 99.25 %, Si = 0.021 %, C = 0.20 %) as very fine powder and silicon (Si = 95.28 %, Fe = 1.82 %, Al = 1.30 %, Ca = 1.01 %).

The reactant mixture was heated (like pressed bricks) at various temperatures in the range of 1100 - 1600^{0}C, under high purity argon and nitrogen protection medium (at 0.3 atm pressure).

Phase determination was performed by X-ray diffraction (SEIFERT Cu cathode) analysis and microstructural observation was conducted using a transmission electronic microscope (JEM - 100 CX).

The X - ray analysis of $MoSi_2$ showed that at 1150^{0}C molybdenum silicides are not formed. Only Silicon and Molybdenum are present at this temperature.

The forming process of the molybdenum silicides begins at 1200^{0}C.

At 1300^{0}C $MoSi_2$ forms together with Mo_5Si_3 and $MoSi_{0.65,}$ and at 1350^{0}C $MoSi_2$ is formed with high

intensity, and Mo_5Si_3 and $MoSi_{0.65}$ keep producing, too.

At 1400^0C and 1450^0C, $MoSi_2$ is produced with very high intensity, the Mo_5Si_3 with high intensity, without the presence of $MoSi_{0.65}$.

For a temperature up to 1500^0C, only $MoSi_2$ is produced with very high intensity.

5. REFERENCES

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