



	Experiment title: High-pressure melting study of vanadium, niobium, and tantalum	Experiment number: HS-4049
Beamline:	Date of experiment: from: 13/05/2010 to: 18/05/2010	Date of report: 12/02/2011 <i>Received at ESRF:</i>
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Report:

Our goal was to study the melting curves of different transition metals. We performed eight independent experiments on Nb and eight experiments on V. Experiments were performed under a variety of pressure-transmitting media covering pressures up to 120 GPa. A summary of the experiments is provided in the table.

Run	Sample	P range (GPa)	T range (K)	gasket	Pressure medium	comments
1	Nb	25.4	2700	Re	NaCl	Being analyzed
2	Nb	42	2900	Re	KBr	Being analyzed
3	Nb	57	3110	Re	NaCl	Melting
4	Nb	11.3	2740	Re	KBr	Melting
5	V	33	2870	Re	KBr	Being analyzed
6	Nb	45.4	2750	Re	KBr	Being analyzed
7	V	27.5	3000	Re	KBr	Recrystallization
8	V	53	4000	Re	KBr	Melting
9	V	90		Re	Al ₂ O ₃	Fail
10	Nb	56.4	3100	Re	Al ₂ O ₃	Phase transition + melting
11	V	64.6	RT	Re	He	Phase transition
12	V	63.9	3200	Re	NaCl	Phase transition
13	V	33	2600	Re	Al ₂ O ₃	Possible chemical reaction
14	Nb	24	3500	Re	KBr	melting
15	Nb	120	4000	Re	MgO	melting
16	V	120	3300	Re	MgO	Being analyzed

The criterion used to determine melting was the observation of diffuse x-ray scattering signal and the disappearance of Bragg peaks from the solid. In many cases, before it happened we observed a strong recrystallization of the samples. P-T conditions in the studies were determined using pyrometry, ruby fluorescence and known EOS. In some cases a run-away heating took place being difficult to determine the temperature at which structural changes happened.

During the experiments we have faced some problems, for instance the possible occurrence of chemical reactions between the sample and pressure medium. Despite the numerous experimental difficulties we have been able to determine the melting point of Nb and V at several pressures. Apparently the melting slope is slightly higher than determined in previous studies. However the results need confirmation of additional experiments. In particular, special care should be taken in future experiments to avoid chemical reactions. In addition to melting, we also observed hints of the existence of a new HP-HT phase in Nb. The new phase apparently has an orthorhombic structure. Since in a second experiment at similar conditions the phase transition was not observed its existence needs to be confirmed from additional studies. The new phase is quenchable to ambient pressure, which could facilitate the identification of its structure. Also additional studies are requested to clarify how oxidation or carbon migration could affect the results.