

# **METALLOTHERMIC SHS REACTIONS INVOLVING NIOBIUM OXIDE**

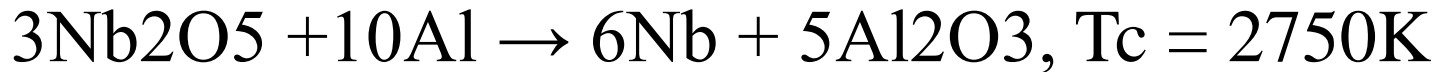
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# CHEMICAL SCHEMES OF SHS FOR CAST ALLOY Nb-Si( Hf, Ti, Al)

## Preparation of Nb:



## Preparation of cast alloy:



# EXPERIMENTAL

## Reagents:

powders of Nb<sub>2</sub>O<sub>5</sub>, Cr<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>; Al, Hf, Si

granules HfAl<sub>3</sub>: d: 0-40, 100-160, 160-300 MKM

## Initial mixtures

- Nb<sub>2</sub>O<sub>5</sub>/Al/Si/Hf/Ti

- Nb<sub>2</sub>O<sub>5</sub>/Al/Si/HfAl<sub>3</sub>/Ti

# CENTRIFUGAL SHS INSTALATION

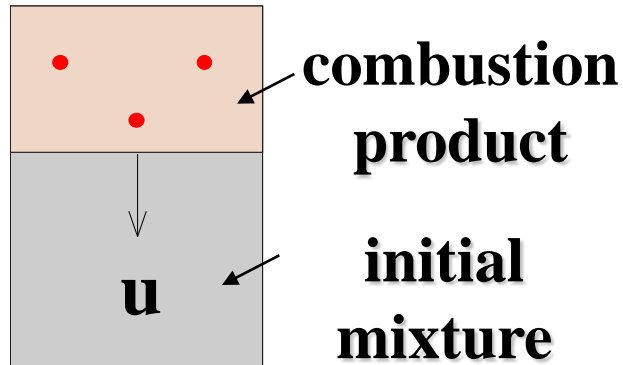


**Overload – 50-500 g**

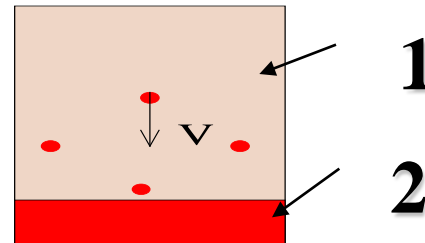
**Mass of initial from 0,5 to 1,5 kg.**

# MAIN STAGES IN SHS OF Nb-Si( Hf, Ti, Al) ALLOY

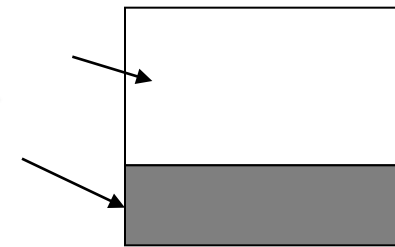
combustion



gravity  
separation



cooling,  
forming of  
structure

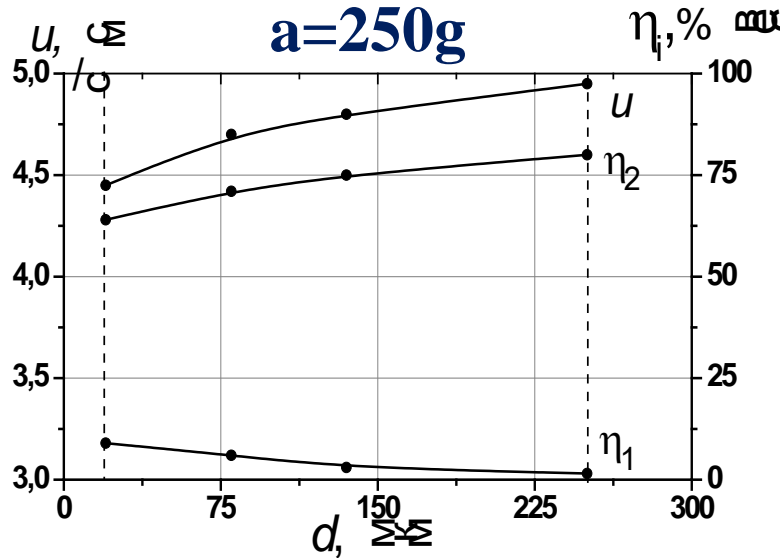


(1) –oxide phase, (2) – alloy

## Characteristic times

- burning time:  $t_1 = H / u$
- gravity separation time:  $t_2 \sim h/v$ ,  $v = (\rho_2 - \rho_1)d_m^2g / 18\mu$
- cooling time:  $t_3 \sim D^2 / a$

# INFLUENCE OF HfAl<sub>3</sub> GRAIN SIZE ON SHS PARAMETERS



$u$ -combustion velocity  
 $\eta_1$ -fullness of mixture sputtering  
 $\eta_2$ -completeness of output alloy  
 in ingot

## Two layered cast SHS product

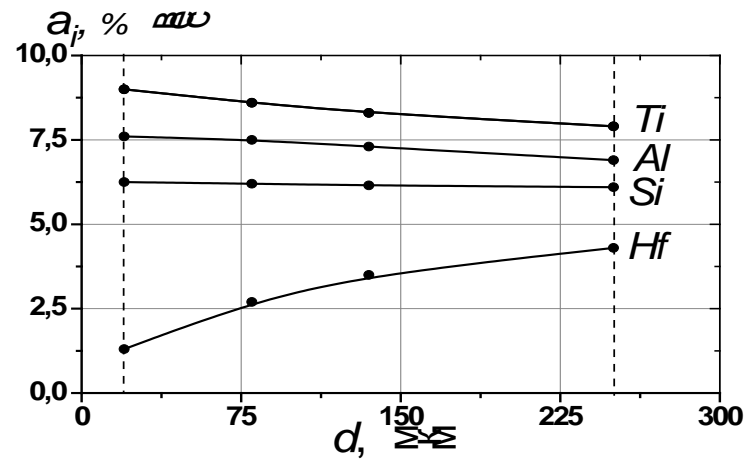
Nb-Si( Hf, Ti, Al)  
alloy



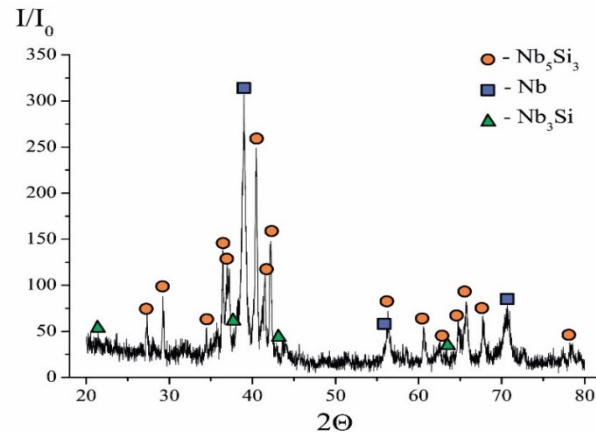
Oxide  
product

# INFLUENCE OF HfAl<sub>3</sub> GRAIN SIZE ON CHEMICAL COMPOSITION Nb-Si( Hf, Ti, Al) ALLOY

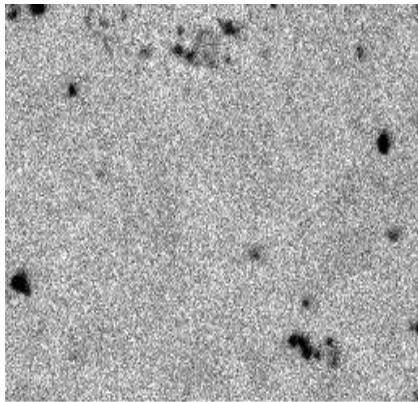
**Nb = 71 - 72%, bec.**



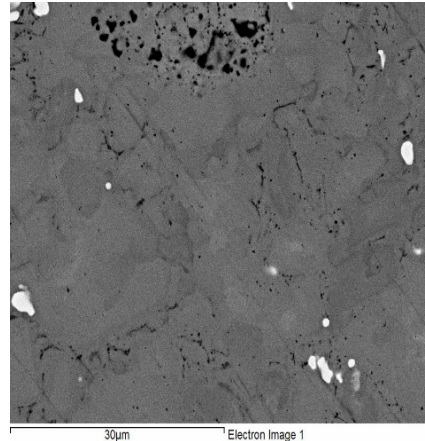
## Phase composition of bottom layer of combustion product



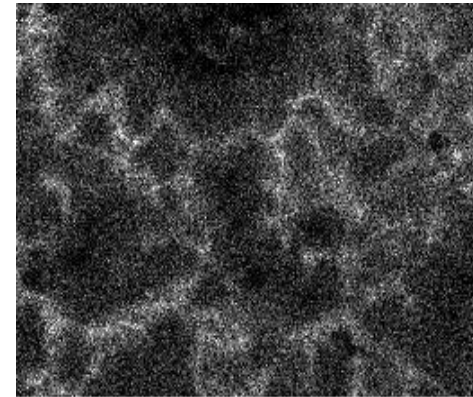
# The map of elements distribution in the Nb-Si( Hf, Ti, Al) alloy



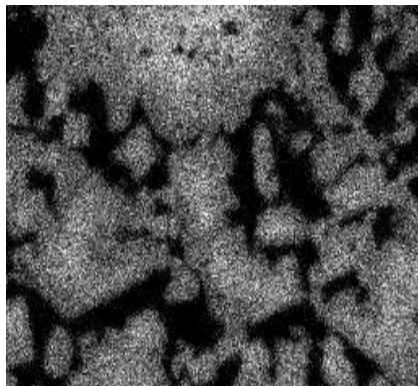
Nb La1



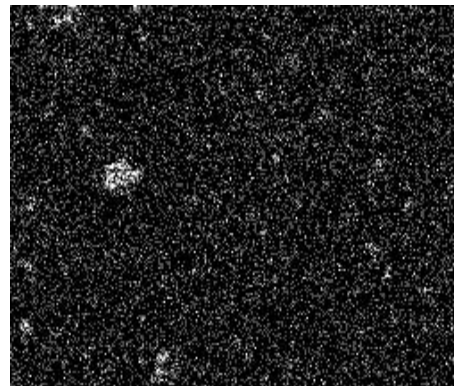
30µm Electron Image 1  
microsection



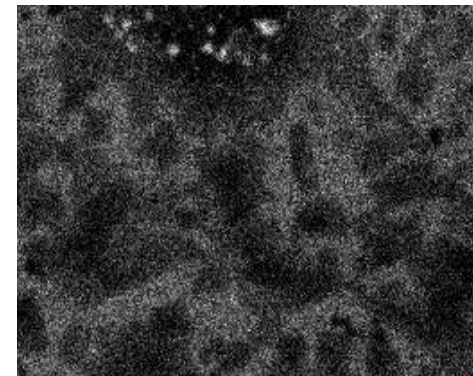
Ti Ka1



Si Ka1



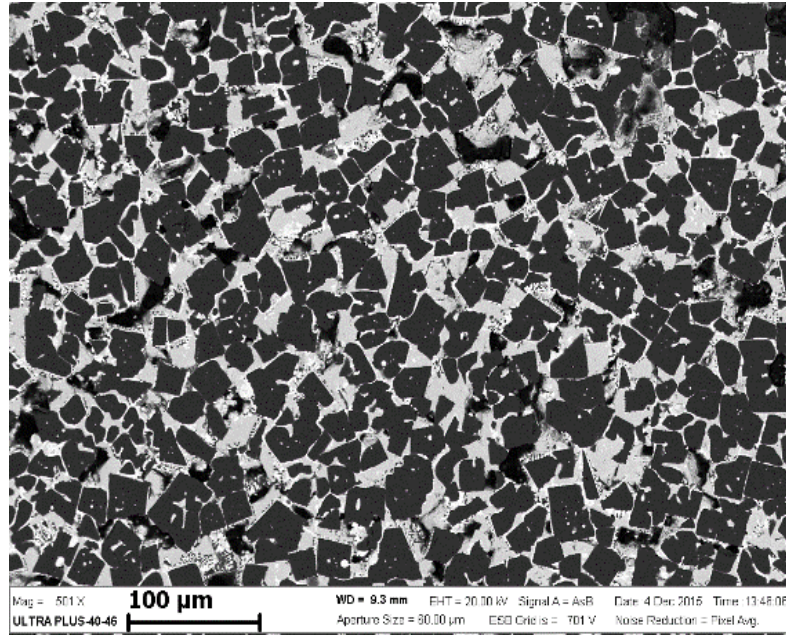
Hf La1



Al Ka1

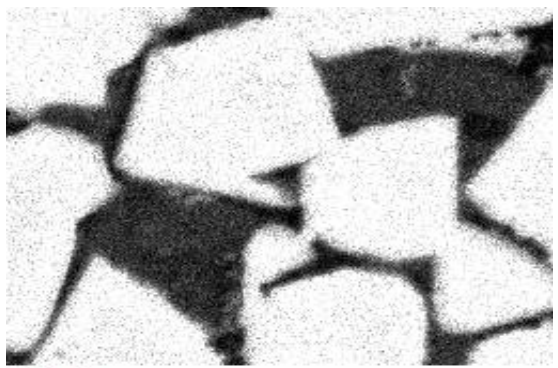


# COMPOSITION AND STRUCTURE OF OXIDE COMBUSTION PRODUCT (LAYER UPPER LAYER)

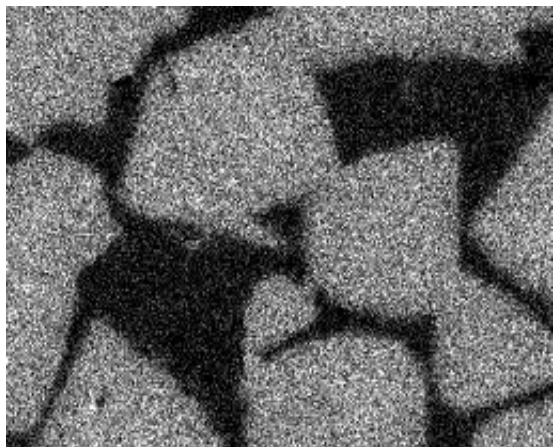


<b>O</b>	<b>Al</b>	<b>Si</b>	<b>Ti</b>	<b>Nb</b>	<b>Hf</b>
<b>48.0</b>	<b>30.3</b>	<b>0.5</b>	<b>6.3</b>	<b>0.9</b>	<b>13.1</b>

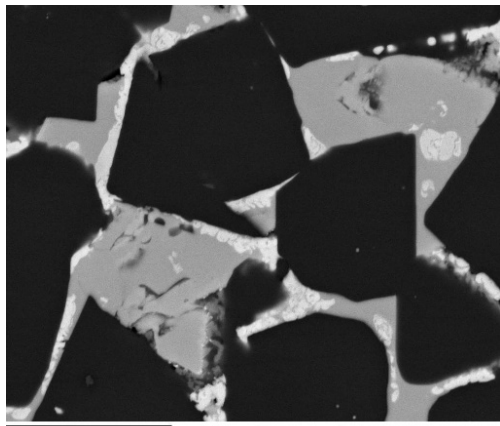
# MICROSTRUCTURE AND ELEMENTS DISTRIBUTION IN OXIDE LAYER



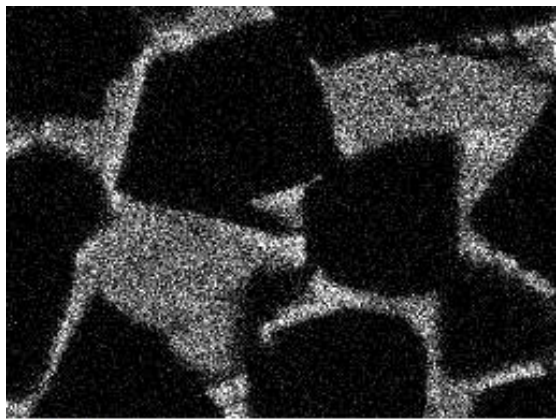
Al Ka1



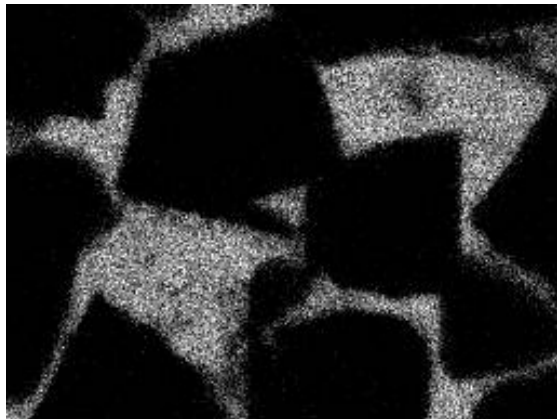
O Ka1



20μm Electron Image 1  
microsection

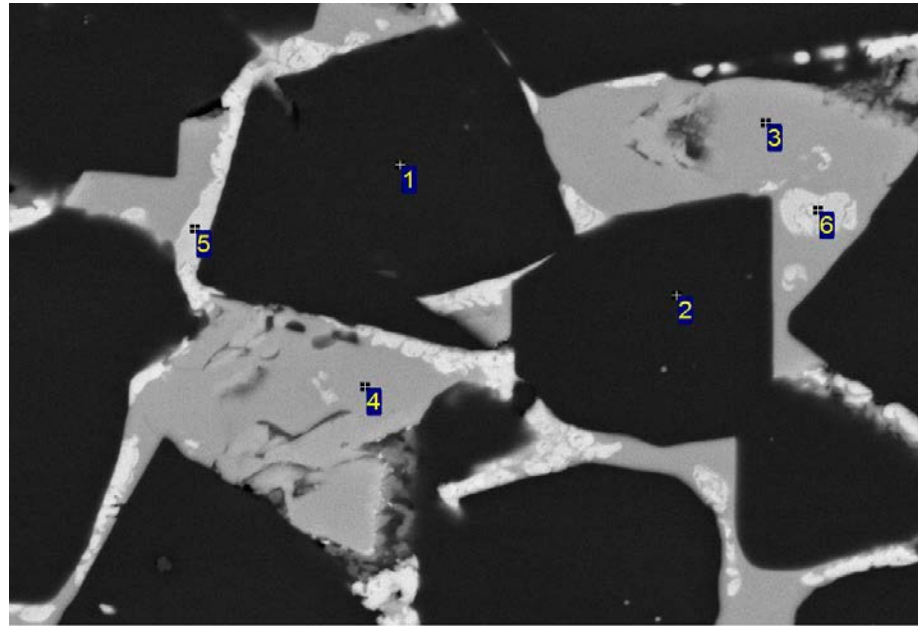


Hf La1



Ti Ka1

# MICROSTRUCTURE AND CHEMICAL COMPOSITION OF STRUCTURAL UNITS IN OXIDE LAYER

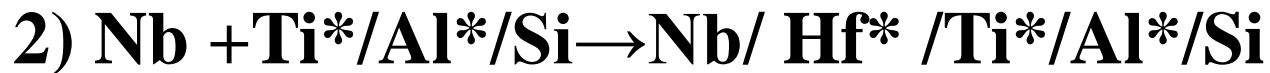
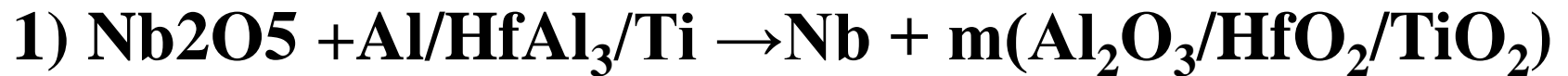


20µm

Electron Image 1

<b>№</b>	<b>O</b>	<b>Al</b>	<b>Ti</b>	<b>Hf</b>
<b>1</b>	<b>54.0</b>	<b>44.4</b>	<b>1.0</b>	<b>0.4</b>
<b>2</b>	<b>53.9</b>	<b>45.0</b>	<b>1.0</b>	<b>0.2</b>
<b>3</b>	<b>33.5</b>	<b>10.7</b>	<b>20.3</b>	<b>33.5</b>
<b>4</b>	<b>33.7</b>	<b>10.4</b>	<b>20.8</b>	<b>32.4</b>
<b>5</b>	<b>28.6</b>	<b>5.0</b>	<b>16.3</b>	<b>48.2</b>
<b>6</b>	<b>27.3</b>	<b>4.5</b>	<b>16.7</b>	<b>49.5</b>

# STUDIES OF CHEMICAL CONVERSION IN COMBUSTION WAVE



# CONCLUSION

- The results demonstrate that centrifugal SHS can be used to fabricate cast niobium silicide based composite materials.
- After combustion, centrifugal forces separate a two-phase products melt on two layer; the top (slag) layer is oxides and the bottom layer is “metallic.”
- The metallic bottom layer has a composite structure, consists of the base elements (Nb, Si, Hf and Ti), and contains three phases: a Nb based phase, Nb<sub>5</sub>Si<sub>3</sub> and Nb<sub>3</sub>Si.
- The slag layer has a two-phase structure: (1) solid solution based on Al<sub>2</sub>O<sub>3</sub> and (2) solid solution based on Hf and Ti oxides.