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POWDER VIA THE ROTATING ELECTRODE PROCESS

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AN INVESTIGATION INTO THE PRODUCTION OF METASTABLE Nb₃Ge
POWDER VIA THE ROTATING ELECTRODE PROCESS

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ABSTRACT

The production of metastable Nb₃Ge powder via the rotating electrode process (REP) employing "splat cooling" was investigated. An electrode capable of withstanding the thermal shock of the electric arc used in REP was produced through powder metallurgy techniques. The effect of various parameters involved in the rotating electrode process was studied in correlation with process control and crystal structure, microstructure and compositional analyses of the powder produced. Superconducting transition temperature measurements were made on the powder both as-produced and after annealing experiments.

INTRODUCTION

The A15 structure in the Nb-Ge system (Fig. 1) was first identified by Carpenter and Searcy¹ in 1956. The compound thought to be Nb₃Ge prepared in bulk form had a superconducting transition temperature (T_C) of about 6°K. Shortly afterwards, Geller² found that the material prepared was nonstoichiometric. Since then, a phase diagram of the Nb-Ge system (Fig. 2) has been published by Pan, et al.,³ showing that A15 Nb₃Ge (also called β -Nb₃Ge) can exist with a range of germanium concentration; from 15 to 21 at.% Ge at various temperatures.

In 1960 Pol Duwez⁴ first developed the "splat cooling" technique, which was later to play an important role in the development of high temperature superconductivity in A15 Nb₃Ge. This technique involves an extremely rapid quench of metal from the molten state. Later studies by Predecki,⁵ et al., and Ruhl⁶ have determined splat cooling rates in excess of 10^6 °C/sec, and, among other effects, the ability to retain high-temperature phases.

In 1965 Bernard Matthias⁷ used the splat cooling method in an attempt to form purely stoichiometric A15 Nb₃Ge. The material he produced had superconducting transition temperatures ranging up to 18°K and evidence indicated that this high T_C was due to the three to one stoichiometry obtained.

In 1973, John Gavalier⁸ reported a new record-high T_C for Nb₃Ge, 22.3°K, obtained by a dc sputtering process. Testari, et al.,⁹ verified Gavalier's sputtering results with a slightly better transition temperature of 23°K. Sputtering involves projecting a high pressure gas, usually argon, onto a composite cathode, in this case made of niobium and

germanium, resulting in deposition of the compound formed by evaporation onto a heated substrate (700-1000°C) held about one inch from the cathode. Sputtering Nb₃Ge requires high gas pressures and low voltages, termed low energy sputtering, making the process diffusion controlled. Deposition rates for sputtering are very slow and only thin films a few thousand angstroms thick can be produced.¹⁰

The search for a more practical method of producing high-T_C Nb₃Ge led Valueva, et al.,¹¹ to chemical vapor deposition (CVD). The CVD technique involves deposition of Nb-Ge compounds by hydrogen reduction of a vapor phase containing Nb and Ge chlorides, with He as a carrier gas in a flow system at atmospheric pressure. As with sputtering, deposition temperatures range from 700 to 900°C. Valueva reported superconducting transition temperatures from 17 to 19⁰K and later, Braginski and Roland¹² reported CVD samples with a T_C of 21.7⁰K.

The advent of sputtering and chemical vapor deposition techniques for preparing high-T_C A15 Nb₃Ge brought about several studies on the parameters affecting the superconducting properties of A15 compounds. It has been found that "Nb₃Ge" prepared by arc melting and other bulk methods is a disordered material, consisting of more than one phase and being Nb-rich, with respect to the three to one stoichiometry desired.¹³ The assumed compositional dependence of T_C, after high-T_C stoichiometric Nb₃Ge had been prepared by splat cooling, has been proven untrue by Testardi, et al.¹⁴ Sputtered films were prepared with ~40% compositional variations (Nb/Ge = 2.2-2.3) and with T_C onsets above 20⁰K. It was further shown that films prepared by sputtering,

with compositions identical to samples prepared by bulk means ($T_c \sim 6^\circ\text{K}$), yielded T_c 's of approximately 14°K .

Further speculation on the dependence of superconducting transition temperature has arisen from studies disclosing the anomalous behaviour¹⁵ of A15 compounds: a compressibility study of V_3Si at room temperature by Blaugher, et al.,¹⁶ has suggested equilibrium vacancies may be responsible for the temperature dependence of superconducting A15 compounds; Rowel and Schmidt¹⁷ discovered a highly disordered material in high- T_c Nb_3Ge films through electron tunneling studies; and numerous studies correlating T_c with electrical resistivity ratio¹⁸ and with lattice parameter behaviour¹⁹ are evidence of some defect in A15 compounds. Testardi supports the view that a universal as-yet unknown microscopic defect is responsible for the behaviour of A15 compounds, and that this defect may be responsible for the existence of metastable A15 compounds at room temperature. Testardi also points out that, among the A15's, defect formation is not an equally serious problem and is worst of all in the consistent production of Nb_3Ge .²⁰

The high current densities, $J_c = 10^5 \text{ A-cm}^{-2}$ at 210 KG, and critical field properties, $H_{c2} = 370 \text{ kG}$ ²¹, makes Nb_3Ge the most promising superconductor known today. Since the metastable compound maintains its crystal structure up to 1000°C , the production of metastable Nb_3Ge in powdered form would be a significant first step in the fabrication of superconducting multifilamentary Nb_3Ge wire.

The objective of this work was to explore the possibility of producing superconducting Nb_3Ge via splat cooling and the rotating electrode process (REP). REP was invented by A. R. Kaufman²² in 1960

and is being used extensively for the production of reactive metal and prealloyed powders.²³ The process involves impinging an electric arc on the face of a rotating consumable electrode, in this case Nb₃Ge, enclosed in a tank with an inert atmosphere. Existing rotating electrode machines employ large tanks such that the resulting liquid particles are cooled by convection in the inert atmosphere. Splat cooling was achieved by positioning a copper cone around the rotating electrode at such a distance that the particles flung off the electrode by centrifugal force were liquid when encountering the cone.

The inherent brittleness of Nb-Ge compounds suggested the largest barrier to this project would be to produce a target to employ in REP that would be able to withstand the thermal shock produced by the electric arc. Such a target was produced and the resulting powder was examined for structure and superconducting properties. Also investigated was the effect of annealing on the Nb-Ge powder produced.

EXPERIMENTAL PROCEDURE

I. Making the Target

Utilizing powder metallurgy techniques the first attempt at producing a target for REP involved the following steps: 1) mixing niobium and germanium powders (Nb powder - 270 + 400 mesh from Wah Chang and Ge powder as-received, 99.999% pure were used) in the atomic ratio 3:1, respectively, on a tumbler with ceramic pellets for 24 hours; 2) isostatic compaction of the mixture in rubber tubes at 100 ksi; and 3) liquid-phase sintering of the compact. Two targets were sintered, one for 15 min. and the other for one hour, at 1300 C in the high vacuum Brew furnace.

Another attempt at making a target utilized steps one and two above (compaction, however, was done with a 3/4" diameter single action die at 100 ksi) but eliminated step three, running the target in the rotating electrode machine as-pressed.

Efforts were then directed towards producing a target composed of the compound Nb_5Ge_3 (M.P. $\approx 2150^{\circ}C$) and Nb, the proportions calculated to achieve an overall Nb/Ge atomic ratio of three. One target attempt tested the idea of drilling holes in a niobium rod and pressing the Nb_5Ge_3 powder into them, hoping that at 15000 rpm the face of the electrode would be melted uniformly.

Nine targets involved mixing and pressing Nb and Ge powders in the atomic ratio 5:3 as described above. One was arc casted in an argon atmosphere at about -10 psig, and the other eight were sintered in the Brew furnace at various temperatures in an argon atmosphere and then arc casted (Table I).

In each case the weight of the sample was recorded before and after sintering and/or arc casting in order to compensate for any elemental germanium loss during these processes. Carpenter²⁴ and Carpenter and Searcy²⁵ have investigated the decomposition pressures for Nb_3Ge and α and β - Nb_5Ge_3 , respectively, and have concluded that effusate vaporized from heated samples of these materials consisted only of germanium. Compensation for Ge losses was made by adding less Nb powder to the reacted samples, after being crushed by hammer and sieved with a 150 mesh screen.

Fourteen more targets were prepared by the following steps:

- 1) mixing Nb and Ge powders in the atomic ratio 5:3; 2) compaction

of the powder in a 3/4" diameter single-action die at 100 ksi; 3) liquid-phase sintering of the compact; 4) crushing the sintered samples by hammer (subsequent ball-milling was done to one sample) and running through a 150 mesh sieve; 5) combining the reacted powdered material with niobium powder to achieve an overall Nb/Ge ratio of three; and 6) compacting the mixture in a 3/4" diameter single-action die at 100 ksi. The 3/4" diameter single-action die was used in this process over isostatic compaction due to the exactness of form achieved, so as to avoid vibration problems at the high speeds employed in REP.

Various sintering times and temperatures were used for the fourteen targets produced (Table I) while trying to achieve an homogeneous material composed of Nb_5Ge_3 . The process employed most often involved sintering the compact at $900^{\circ}C$, slightly below the melting point of germanium, for one hour, then at $1650^{\circ}C$, slightly below the melting point of the lowest melting point compound in the Nb-Ge system ($NbGe_2$), for two hours, and finally at $1850^{\circ}C$ for one hour. Temperatures were determined with an optical pyrometer.

Initially, samples were suspended into the hot zone of the Brew furnace with 5mm dia. tantalum wire. Above $1650^{\circ}C$, germanium reacted with the wire, embrittling the wire with subsequent breakage and furnace difficulties. A process which eliminated sample containment problems involved low temperature sintering ($\leq 1850^{\circ}C$) in an aluminum oxide crucible and subsequent high temperature sintering in a tantalum crucible. This resulted in a reduction in the extent of embrittlement of the Ta crucible, achieved by the reduction in the amount of elemental

germanium by previous sintering. For future experiments, a tungsten barrier within a tantalum crucible may eliminate any crucible contamination, as only the portions of the Ta crucible directly in contact with the sample were contaminated.

One more target attempt involved heat treatment at 1000°C for 235 hours of Nb and Ge powders, Nb/Ge atomic ratio of 5:3 pressed at 100 ksi in a single action die and placed in a graphite tube, which was further enclosed in an argon filled quartz tube. The reacted samples were afterwards crushed with a hammer, sieved through a 150 mesh screen, and blended with Nb powder to achieve an overall Nb/Ge atomic ratio of three.

II. The Rotating Electrode Process

The rotating electrode machine (Figs. 3 & 4) consisted of a high speed air motor producing 10,300 rpm (higher speeds are possible with accurate pulley alignment). A pulley ratio of 1.43 produced an electrode rotation of 15,000 rpm. Built into the machine was a copper cone, supplied with copper cooling coils, located approximately 16.5 cm from a 3/4" dia. consumable electrode fastened to the spindle by means of set screws. The impact angle made by a particle flung off the target during the process was 120° . A nonconsumable tungsten electrode, connected to a generator supplying 30 volts and 0-300 dc amperes, was hand fed into the face of a consumable electrode. Prior to each experiment the cylindrical tank enclosing the electrodes and cone was filled with argon.

The maximum speed available and current intensities from 90 to 500 amperes, straight polarity, were used during experiments. Ini-

tially, most of the Nb-Ge targets produced powder in the form of spheres, indicating convection cooling. Consequently, two more copper cones were fabricated from 5mm thick sheet to lessen the target-to-substrate distance and obtain splats. The first had a target-to-cone distance of 10.2 cm and an impact angle of 110° ; the second, a distance of 4.6 cm and an impact angle of 125° . The surface condition of these cones was altered by sanding or sandblasting for some experiments. The brief duration of REP experiments produced no appreciable heating of these cones and cooling was not considered, as Pihl²⁶ has reported a negligible difference in splat cooling rates between substrates at room temperature and at -196°C .

The splat cooling rate produced by the rotating electrode machine was not measured directly; however, examination of the microstructure of REP powder presented indirect qualitative evidence of the rate of the liquid quench: Ramachandrarao, et al.,²⁷ have correlated grain sizes in splat cooling (0.1 - 10 μm) with splat thickness, directly associated with cooling rate.

III. Annealing REP Powder

All samples were enclosed in quartz tubes after evacuation and backfilling with argon to a -5 psig pressure. Annealing was done in a Honeywell electric furnace at 950°C , in correlation with a report by Sweedler, et al.,²⁸ that $\text{Al}_5\text{Nb}_3\text{Ge}$ maintains its crystal structure below 1000°C . For targets 10, 15, 18 and 21, an initial one hour anneal was performed, followed by a six hour-50 min. anneal, during which the temperature of the furnace was kept to within 5°C of 950°C . Powder from target 26 was annealed at 950°C for four hours.

IV. Analytical Techniques

Optical microscopy was used to determine the grain structure and homogeneity of the powder produced. All samples were etched with a solution of equal volumes of H_2O , H_2SO_4 , and HF, with a few drops of H_2O_2 . A polaroid MP3 camera was used to photograph the powder at low magnifications.

X-ray diffraction employing the Cu K radiation was used to determine crystal structures. Scans were generally made from 2θ values of 20° to 110° and all samples were x-rayed in powdered form, but of no standard size.

Scanning electron microscopy and EDAX (Energy Dispersive Analysis of X-rays) were used to determine the compositions of phases present.

Superconducting transition temperature measurements were made by the inductive method. This method incorporates the principle known as the Meissner effect, by which magnetic flux is expelled from within a superconductor in the presence of a magnetic field.

The inductive method requires no electrical contacts to the sample, being placed inside a coil, and so lends itself readily to measurements of irregular shapes (e.g. powder).

RESULTS AND DISCUSSION

I. Initial Target Attempts

The targets sintered at $1300^\circ C$ consisted, according to x-ray diffraction data, of cubic Nb_3Ge , tetragonal Nb_5Ge_3 and some pure niobium. The first of these, sintered for 15 minutes, broke apart instantly upon impingement of the electric arc on the target face. A tungsten ring, fastened to the electrode holder, was used to strike

the arc for the second target, sintered for one hour, in order to relieve the target of the high current necessary to initiate the arc. The target began to glow red, lasting for about five seconds, before breaking up.

The failure of the Nb-Ge compounds to withstand the thermal shock of the electric arc led to a next attempt employing the elemental strengths of niobium and germanium. While some powder was produced in REP, this as-pressed target (atomic Nb/Ge = 3) also broke apart and germanium effusate was afterwards noted on the surrounding copper substrate and tungsten ring. X-ray diffraction analysis of the powder showed approximately equal amounts of Nb_3Ge and tetragonal Nb_5Ge_3 to be present.

A cast Nb rod with sintered Nb-Ge powder (at $950^{\circ}C/1$ hr. then $1500^{\circ}C/1$ hr.) pressed into six drilled holes was next tried in order to compensate for the great difference in melting temperatures of niobium and germanium ($2468^{\circ}C$ and $937^{\circ}C$, respectively), and yet again employ the element strength of niobium. Uneven melting occurred on the target surface, the powder being melted away quickly and the niobium almost not at all: the cause for this result was a great difference in thermal conductivity between the cast state, in which the heat from the arc was transferred through the mass of the target and into the spindle too quickly to melt the face, and the powdered state, where, as-pressed, there existed point contact between spheres.

Attempted next was a target composed of niobium powder and arc cast Nb-Ge powder, previously mixed in the atomic ratio 5:3, respectively. The presence of the higher temperature Nb-Ge compounds

in the arc cast sample was verified by γ -ray diffraction. With 240 dc amperes being used in the rotating electrode machine, in less than 10 seconds, a considerable amount of powder was made, predominantly in the form of flakes (splats). X-ray diffraction showed this powder to have the A15 structure with a small amount of a second phase, tetragonal Nb_5Ge_3 . The poor thermal conductivity throughout the mass of the target, providing localization of the arc heat on the target face, along with the good elemental strength of niobium were believed responsible for this success.

II. Optimization of Target Formation

Work next concentrated on repetition of the above results and on obtaining a reacted Nb-Ge powder with a homogeneous Nb_5Ge_3 composition. The limited time in which reaction between powder particles was allowed in REP suggested this would be necessary to achieve an homogeneous REP powder. Table I displays all of the heat treatments attempted, in chronological order, along with remarks on subsequent effects in REP powder production.

Procedures involving sintering with and without subsequent arc casting were used, attempting to obtain a material homogeneously Nb_5Ge_3 . In no case did x-ray diffraction reveal such a result; all samples consisted of two or more compounds in the Nb-Ge system. However, the higher currents necessary to avoid electrode disintegration under the electric arc in REP for samples sintered at 1850°C suggested that more of the higher melting point compounds were being formed by sintering. This conclusion is based on the hypothesis that there is a limited time period in which the target surface must be melted,

after which the electrode will disintegrate due to thermal shock produced by the transfer of heat through its mass; and that this time is related to the melting point of the material, higher melting points requiring higher currents. Three hundred amperes were inadequate in producing splats for sintered targets 13 - 23, whereas 210 amperes were excessive for arc cast samples.

The germanium lost during arc casting may have prevented the formation of the Ge-rich compound, Nb_5Ge_3 . Previous sintering considerably reduced the quantity of germanium vaporized and prior compensation for this effect may solve the problem of obtaining an homogeneous Nb_5Ge_3 material by arc casting.

While it is believed sintering at higher temperatures (2000°C) would also produce an adequate material, the long times involved, sample containment, and furnace contamination, as germanium effusate was deposited on the furnace walls after all heat treatments above 1650°C at atmospheric pressure, make this a more difficult procedure.

The sample sintered at 1000°C for 235 hours consisted, according to x-ray diffraction, of hexagonal Nb_5Ge_3 . A small amount of powder was produced from targets 24 and 25, for which 300 amperes was inadequate, causing target disintegration. Over 500 amperes melted target 26 in seconds, forming thin and thick irregular splats, long strands of conglomerated splats, and tiny spheres. X-ray diffraction revealed the REP powder to consist of hexagonal and tetragonal Nb_5Ge_3 , with a small amount of the A15 structure. Powder from target 26 was subjected to a four hour anneal at 950 C, resulting in no change in structure or composition, according to SEM and EDAX analyses (Fig. 5).

This target construction, from these results, is unsuitable for metastable Al₅Nb₃Ge powder production.

III. REP Parameters

The effects of speed of rotation, current intensity ($V=\text{constant}$), impact angle, target-to-substrate distance and substrate surface condition were studied while employing targets two through twenty-six in the rotating electrode machine. Hand operation of the Lungsten lip electrode provided coarse control of the arc distance, and correspondingly the localization of heat input into the target, resulting in many discrepancies in Table I (ie. success for one target and failure for another while employing identical conditions).

Current intensity proved to be a major factor concerning the size and shape of powder produced, and was solely responsible for keeping the target intact during the process. Below a certain current intensity the target would disintegrate; above this value an irregular shaped powder (Fig. 6b), along with smaller spherical particles, was produced (apparently, only in the smaller particles are surface tension forces able to achieve a spherical form before freezing); increasing the current further produced splats (Figs. 6a & 7a), as the particles flung off the electrode remained liquid until encountering the copper cone; an excessive current intensity produced thick splats (Fig. 7b) due to an extremely rapid melting rate and larger particle formation.

With the use of an excessive current intensity another parameter, target-to-substrate distance, greatly affected the type of powder produced. With increased fluidity of the liquid encountering the

substrate, decreasing this distance promoted splatting action, as noted for target 16.

The range of control with these two parameters is fortunate, for optical micrographs (Fig. 8) show conclusive evidence that thin splats provide the fine grained structure responsible for the high current carrying capacity of superconducting Nb_3Ge . Thicker splats and convection cooled particles are characterized by a larger grained morphology (Fig. 9). The target-to-substrate distance is an important parameter that should be optimized, to provide predominantly thin splats, once target processing and current intensity have been established.

The range of powder sizes and shapes obtained from REP experiments suggests some control of superheating, a parameter affecting the degree of reaction between powder particles, is available in REP. An upper limit, as centrifugal force expels the liquid phase from the target face as soon as it exists, may decide the fate of using REP for A15 Nb_3Ge powder production. SEM and EDAX analyses of powder from target 18 (Fig. 10) indicate the amount of superheat was insufficient in this case, as an incomplete reaction resulted in more than one phase to be present, along with an eutectic structure. Transition temperature curves of targets 10, 15, 18 and 21 comparatively indicate 10, from which thick splats were obtained, to be the most homogeneous composition.

The unavoidable formation of a second phase, Nb_5Ge_3 , places a question mark on whether stoichiometric A15 Nb_3Ge can be obtained with REP, for this compound depletes the A15 phase of germanium.

Only a Ge-rich target might achieve the stoichiometry desired, and such compensation might more dominantly promote formation of Nb_5Ge_3 .

Substrate surface condition proved to be of major influence regarding contact between liquid particles and the substrate. Increasing the surface roughness, by sandblasting or sanding, improving sticking such that splats required scraping to be removed from the cone. This result represents a higher heat transfer rate at the interface of the two surfaces; however, this condition destroyed continuous operation of the process as a layer of splats quickly formed on the copper cone.

Neither impact angle nor the speed of rotation were varied appreciably during the experiments and were not considered critical parameters. However, the impact angle should not be too large ($\geq 150^\circ$) in order to assure rapid completion of contact of a liquid particle and is only limited in sharpness such that powder removal allows continuous operation (i.e. in a cylindrical form powder would collect at the bottom). The maximum speed allowed by the equipment should be used to promote splatting action.

IV. T_c Measurements and Annealing Effects

No sample achieved a T_c above 7°K . This result must be attributed to composition, as EDAX analyses (Fig. 10) proved the A15 powders to consist of about 15-19 at.% Ge (phase 2), for which no appreciable T_c has ever been recorded.²⁹ A 7.8 hour anneal at 950°C homogenized the material (Fig. 11), retaining the A15 structure according to x-ray diffraction, but resulted in a uniform composition of ~19 at.% Ge.

CONCLUSIONS AND RECOMMENDATIONS

1. The rotating electrode process appears suitable for the production of Nb-Ge A15 powder from targets pressed from niobium and tetragonal Nb_5Ge_3 powders with an overall Nb/Ge atomic ratio of three.
2. Further refinements of parameter control in the rotating electrode machine are necessary. Such parameters include: 1) precise control of arc distance between the two electrodes; 2) variable control of the target-to-substrate distance; 3) means of removing particles adhered to a sandblasted surface, a condition found to achieve the most efficient splat cooling; and 4) easy and accurate regulation of electrode rotation speed.
3. Conclusive evidence to support the use of REP to obtain metastable Nb_3Ge was not obtained; however, it is believed that a target produced by high-temperature sintering might achieve this result, with adequate Ge compensation for losses.
4. The formation of a second phase, Nb_5Ge_3 , appears inevitable and further emphasizes the need for Ge compensation.

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APPENDIX

A guide for operation of the rotating electrode machine (refer to figure 12.)

1. Install consumable electrode and align axially with a mueller gage.
2. Secure frontcover of machine and begin filling tank with argon: complete Ar atmosphere requires 10-15 min., the air being expelled from the tank through outlet at top.
3. Connect air motor to air pressure line (90 psi) and run at low speed while checking lubricator and adding 5-10 drops of Chevron RPM Handy Oil to bearing's box.
4. Connect ground wire from carbon brush to positive ground. An insulator plate and the carbon brush isolate bearings from electrical circuit.
5. Connect hose from cooling source to copper coils on copper cone if desired.
6. Place generator on straight polarity and adjust the current and start-adjustment to desired values. Place operator control on remote for foot pedal operation.
7. Plug in light and, if argon filling is complete, begin operation by: a) start motor -- gate valve open; b) lift welding lense for primary positioning of tungsten tip electrode; c) replace lense and press foot pedal all the way down to start arc (if arc does not occur feed in further the tungsten electrode).

Notes on operation and maintenance:

1. If target breaks up stop process immediately.
2. Air motor requires occasional maintenance (see guide).
3. Tungsten tip requires occasional grinding.
4. Bearings can withstand speeds to 32,000 rpm.
5. Rotation speed of consumable electrode may be measured with a photo-tachometer, taking reading off lower pulley (fan belt system provides a pulley ratio of 1.43).

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TABLE I

Target No.	Sintering Temp. (°C)	Parameters Time (min.)	Arc Casted	Current used in REP (dc amperes)	Cone used	Remarks on powder production
1	----	----	X	240	1	many splats
2	1100***	60	-	220	1	few splats; irregular part.
3	1700	10	-	180	1	same as '2' but smaller
4			-	120	1	few splats
5	1050	60	X	120	1	same as '4'
6	1600	10	X	90	1	electrode broke up
7	1100	60	X	150	1	sm. irregular part.
8			X	150	1	electrode broke up; some part.
9	1100	60	X	210	1	few splats; irr. part.
10	1650	60	X	210	1	thick splats
11	1000	60	X	180	1	electrode broke up
12	1650	105	X	180	1	fine spheres; few splats
13	950	60	-	180	2	same as '13'
14	1650	60	-	225	2	same as '13'
15	1850	60	-	255	2	few splats; irr. part.
16	850	60	-	300	2	same as '15'
17	1650	60	-	300	2	same as '15'
18	1850	15	-	300	3*	mostly splats-stuck to cone
19	900	60	-	300, 270	2*	few splats
20	1650	120	-	300	2*	few splats
21	1850	60	-	270	2**	some splats
22	925	60	-	270, 255	4	same as '19'; elec. broke up
23	1650	120	-	270	4**	some splats
	1850	60	-			
24			-	300	4	irr. shapes; elec. broke
25	1000	235 hrs.	-	300	4*	some splats; elec. broke
26			-	500	4**	splats and sm. spheres

*cone was sandblasted
 **cone was sanded with 600 grit.
 ***two-step sintering; (i.e., 1100 C/1 hr.,
 then 1700 C/10 min.)

cone parameters:

1
 2
 3
 4

Distance to target

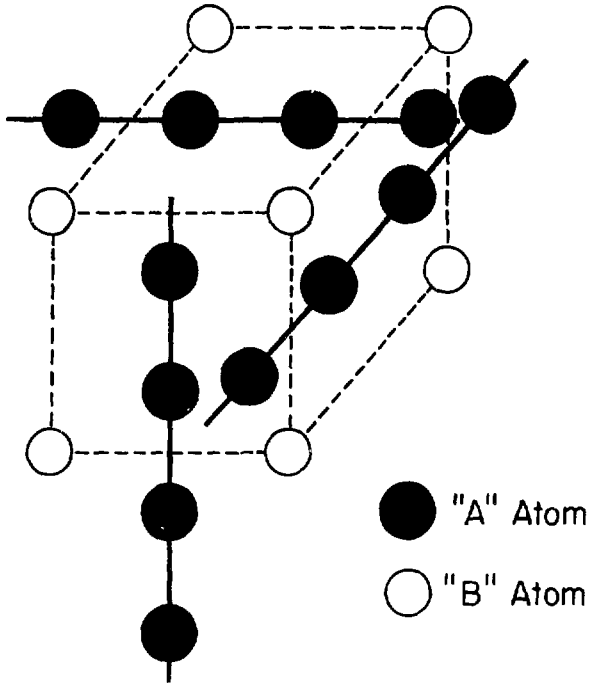
16.5 cm
 10.2 cm
 4.8 cm
 4.6 cm

Impact angle

120°
 110°
 90°
 125°

FIGURE CAPTIONS

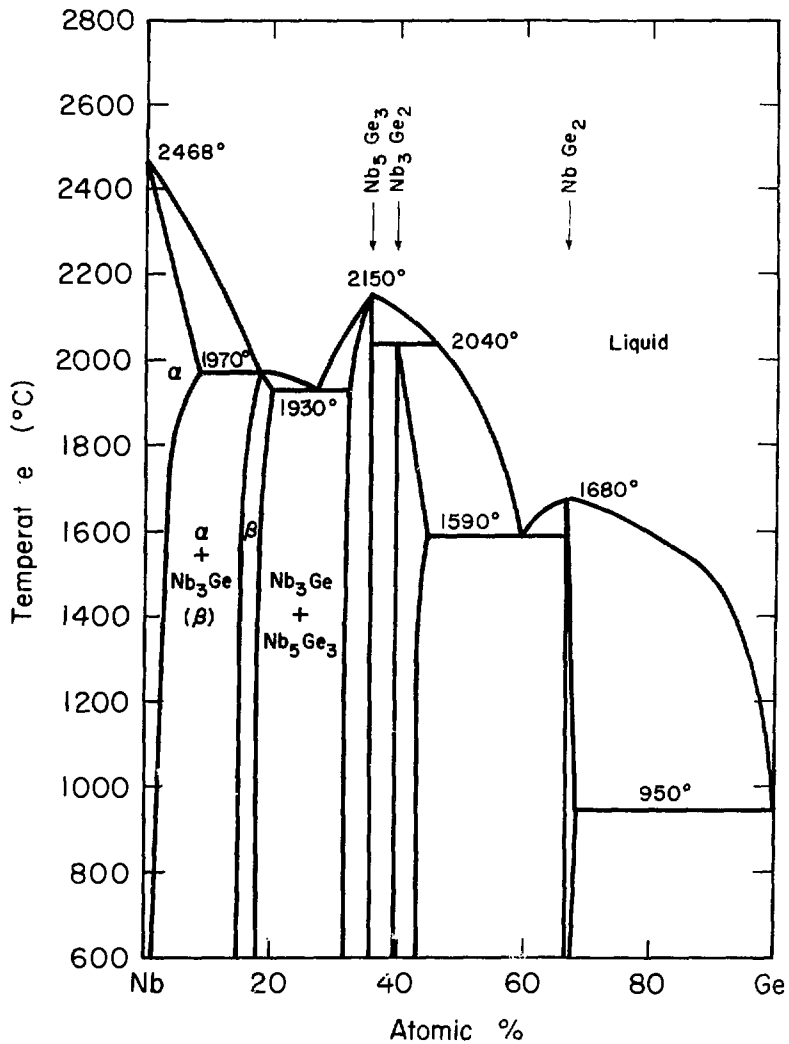
1. Al₅ Crystal Structure.
2. Phase diagram of niobium-germanium system.
3. Frontal view of rotating electrode machine.
4. Side view of rotating electrode machine.
5. SEM micrograph with EDAX display from target 26.
6. a) Splats and b), irregular shapes produced by rotating electrode process from Nb-Ge targets 21 and 26, respectively.
7. a) Thin splats and b), thick splats produced by REP from Nb-Ge targets 18 and 10, respectively.
8. Optical micrographs characterizing inhomogeneity and fine-grained morphology of splat cooled particles.
9. a), b) and c) Morphologies of convection cooled particles from REP; note porosity present in all types of particles.
10. SEM micrograph with EDAX display from target 19.
11. SEM micrograph with EDAX display of powder particle from target 19 after a 7.8 hour anneal at 950 C.
12. Schematic diagram of rotating electrode machine: 1) Air motor; 2) fan belt-pulley system; 3) bearings; 4) carbon brush; 5) consumable electrode; 6) tungsten tip, nonconsumable electrode; 7) cylindrical tank; 8) copper cone equipped with cooling coils; 9) gas outlet; 10) viewing port; 11) light; 12) machine base; 13) gas inlet; 14) insulating plate.



A15 structure
formula type A_3B

XBL 763-2568

Fig. 1



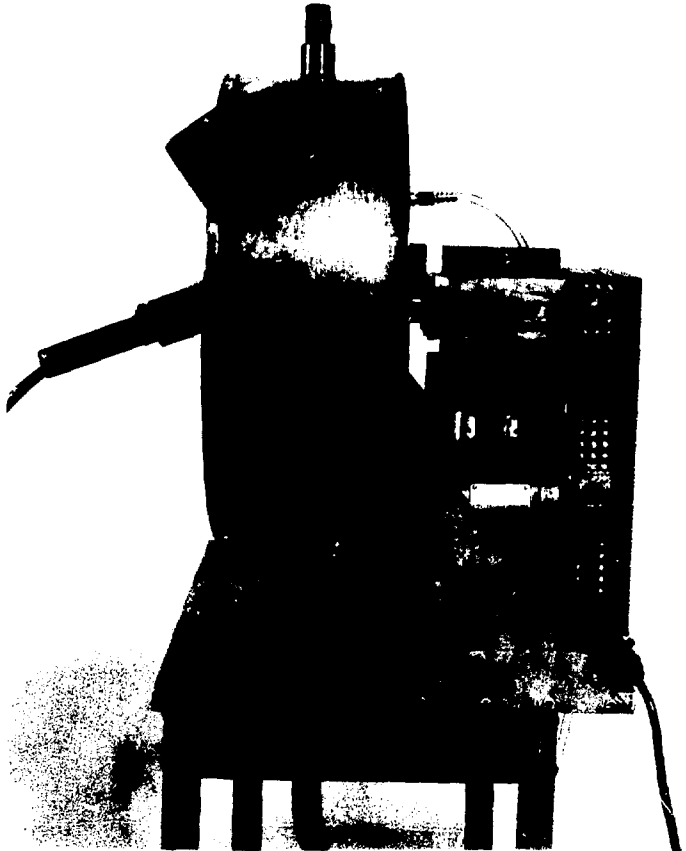
XBL 763-2565

Fig. 2



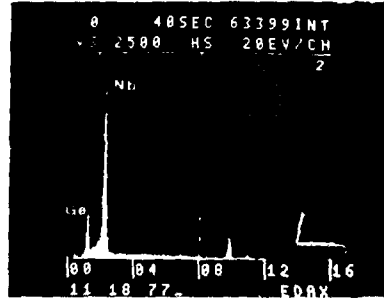
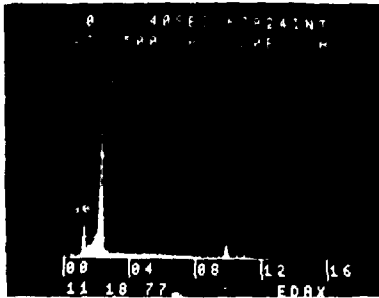
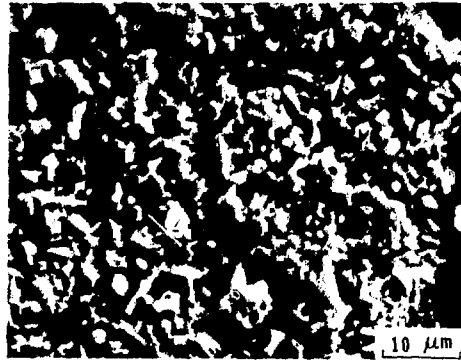
XBB 750-8196

Fig. 3



XBB 750-8195

Fig. 4

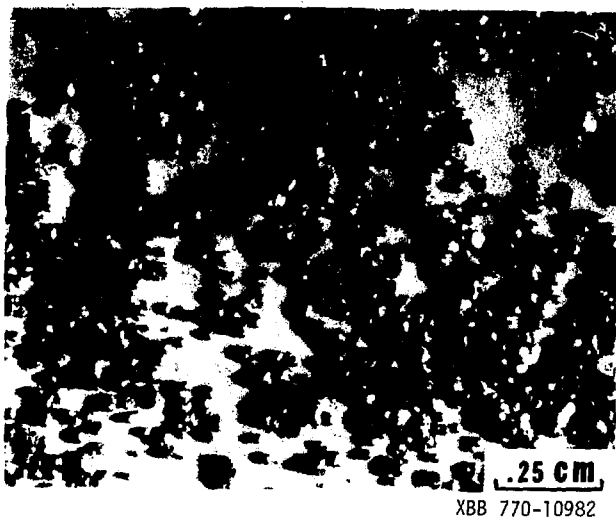


XBB 770-11730

Fig. 5



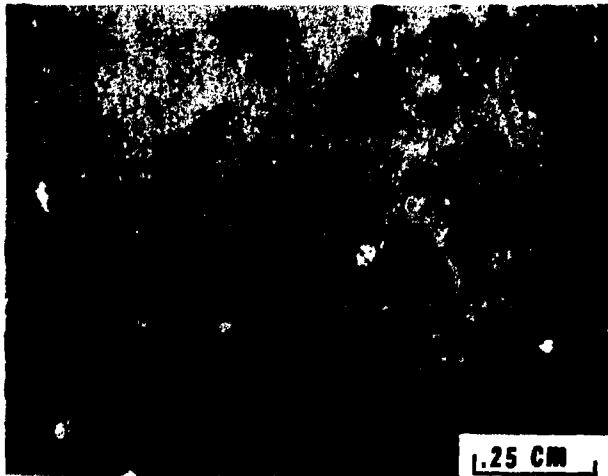
a



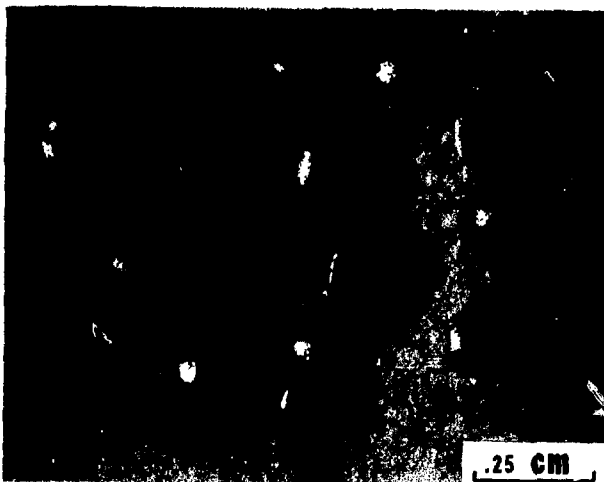
b

XBB 770-10982

Figs. 6



a



b

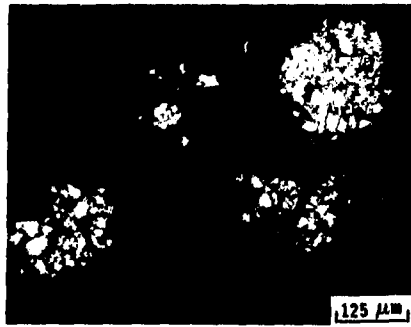
XBB 770-10978

Figs. 7

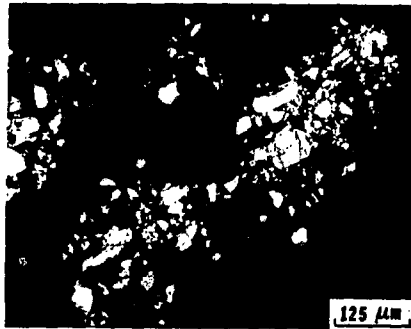


XBB 770-10983

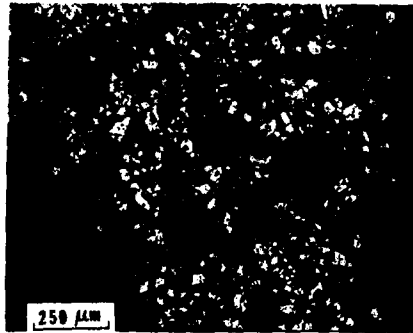
Fig. 8



a

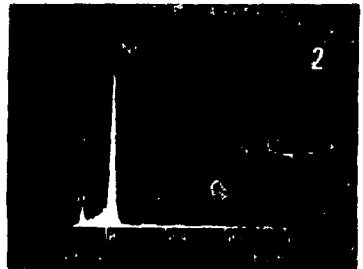
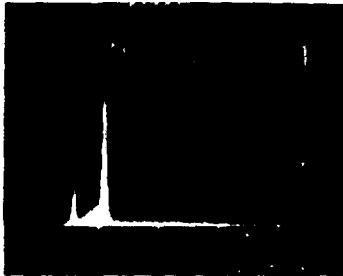
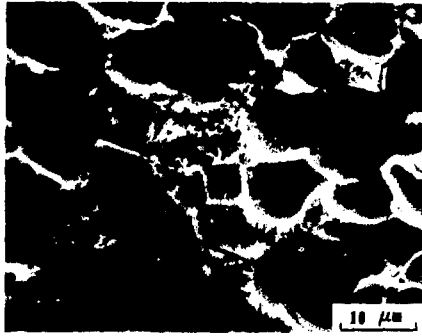


b



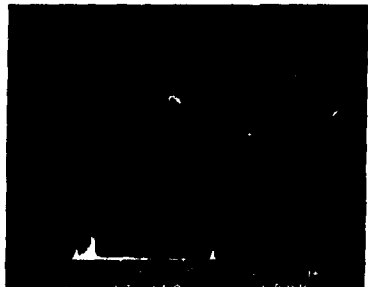
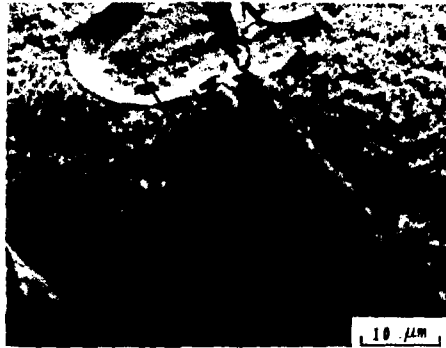
c XBB 770-10980

Figs. 9



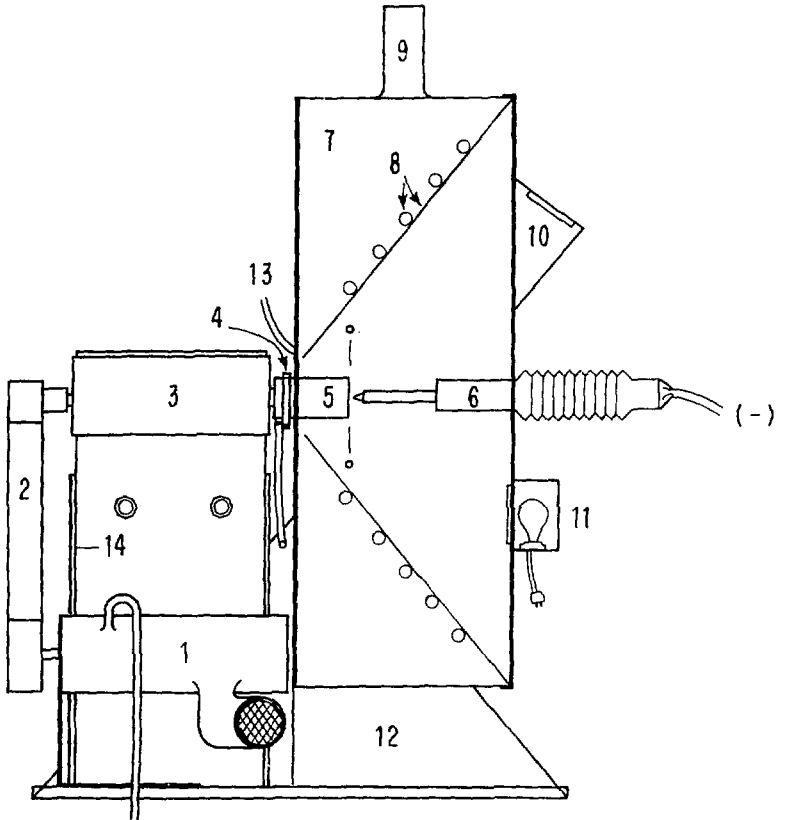
XBS 770-10981

Fig. 10



XBB 770-11493

Fig. 11



XBL7710-2128

Fig. 12