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(FRANCE) G ANDERSON 30 NOV 87 R/D-5002-PH-01
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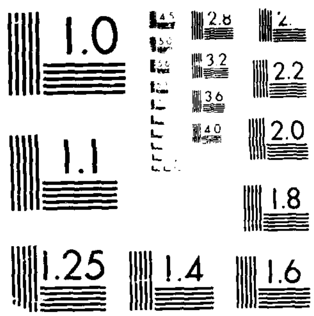

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Date : Nov. 25th, 1987

STUDY ON IRON DISILICIDE THERMOELECTRIC GENERATOR

INTERIM REPORT NOV. 30TH, 1987

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Contract n° DAJA 45-87-C-0057
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The study has 2 parts a bibliographical analysis (Prof. Hubert SCHERRER) and thermal flux prototype (Dr. Philippe SCHLICKLIN).

I - BIBLIOGRAPHY

Using the accessible data bases, we are gathering papers from many countries (U.S.A. , Japan, Soviet Union, France, Germany etc...). 50 publications have retained our interest of which about 10 concern the thin film material.

The first interesting information that we have got out of these papers concerns the phase diagram of the system Fe Si. According to temperature, iron disilicide exists in two forms alpha and beta.
The form alpha, stable at high temperature belongs to the quadratic system type D'4h (PHRAGMEN, 1926 ; ARENSON, 1960).

This form presents a systematic deviation of composition due to the presence of lacunas in the iron sub-lattice (DISORENKO, GEL'D and DUBROWSKAYA, 1964). Its properties are well known and its preparation is easy ; the alpha form is obtained in the usual conditions of silicide formation, and althought it splits below around 950 °C as :

$$\alpha \rightarrow \beta \quad + Si \qquad \text{(I)}$$

an appropriate rate of cooling allows one to obtain the alpha form in the metastable state at room temperature.

The low temperature beta form is stoichiometric and splits rapidily above 970 °C as :

$$\beta \rightarrow \alpha \quad + Fe\ Si \qquad \text{(II)}$$

The transformations (I) and (II) were studied in detail because of the complete change in the electrical properties. The beta $FeSi_2$ being a semi conductor, while the alpha $FeSi_2$ is of the metallic type (BOL'DBERG, LIPATOVA and GEL'D, 1964 ; BIRKHOLZ and SCHELM, 1968).

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These phase transformations were characterized using the Mossbauer-effect (GENIN, LE CORRE, 1972) and this technique will be developed later.

Only the beta $FeSi_2$ is interesting from the point of view of thermoelectric properties. The elaboration of single crystal of beta $Fe\ Si_2$ was presented by WANDI, DUSAUSOY, PROTAS and ROQUES (1968), using chemical transport by iodine. This technique is not well suited to obtain large size single crystals. Thus it is more interesting to use polycrystalline materials obtained by annealing at 800 °C of alpha $FeSi_2$.

Both N type and P type materials can be obtained by doping. The replacement of a part of the iron atoms by an element such Co or Ni gives the N type material. The substitution of silicon by an element of the III group (Mn, Al) leads to the P type material.

Moreover the technique of elaboration presents no special difficulties and seems to have a low cost. The material preparation implies only the reaction in the molten phase of the starting materials, which have a medium level of purity (99.5 %)
The material is then ground down to a size of 3 micrometer, isostatically cold pressed and annealed under vacuum at 200 °C.

The sintering is done at 1175°C and the transition from alpha-phase to beta-phase is obtained by annealing at 800 °C for several days. The doping materials are added to the starting materials.

The bibliography reports values of the factor of merit Z of less than $1 \times 10^{-3}\ K^{-1}$.

Some papers are on amorphons thin film made by "Ionized Cluster Beam").

The factors of merit that are claimed are very high but in the absence of any measured value of thermal conductivity, the caracteristics require checking.

All the references selected will be given and discussed, the above description will be developed and detailed in the final report.

II - THERMAL FLUX PROTOTYPE

The first objective of the experimental work is to verify the characteristics of commercialy available material. The Japanese company KOMATSU ELECTRONICS LTD sells U-Shaped couples of $FeSi_2$. 24 couples are now in our laboratory.
Each leg of the couple is made of one type (N or P) of material and the junction is placed directly into a flame.

Being almost impossible to measure the hot junction temperature in the flame and to evaluate the heat flux going through the material betwen hot and cold junctions, we have designed an experimental assembly, suited to mesure these values.

The assembly comprises a cylindral electrical heater, several $FeSi_2$ couples are placed with their hot junctions at the periphery of the heater.

The main problem is to obtain a good thermal contact for the hot junctions. If not, there is an important temperature drop between the hot source and the hot junction of the couple leading to wrong values of the characteristics (underestimation of the thermal conductivity and the SEEBECK coefficient).

Our first attempt used a molded ceramic sleeve around the cylindrical heater which included the hot junctions of the $FeSi_2$ couples but the contraction of the ceramic material gave bad thermal contact.

We will now try a sleeve made of heat resisting steel. We are studying the best way to measure the temperatures as acurately as possible the main problem being to maintain an electrical insulation between the metallic sleeve and the hot junctions.

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