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Name of Contractor: U.S. Automation Company

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Effective Date of Contract: 89 July 19

Expiration Date of Contract: 90 February 18

Reporting Period: From Beginning of Contract To The End

Principal Investigator and Phone No: Daniel J. Borodin
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Short Title of Work: High Temperature Superconductor
Production Using ... Externally
Induced Shear.

As a result of Phase I effort (covered in the enclosed text) encompassing design of apparatus, interpretation of recent empirical data, and because of the addition of new conceptual ideas, <u>OPPORTUNITY</u> now exists to attain the following new objectives: -

1. Configuration for a continuous high J_c flexible HT-SC cable.
2. Continuous high speed manufacturing pilot line to produce item 1.

Both of the above objectives will be achieved by the use of apparatus depicted on pages 9 to 14 with added attachments.

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PHASE I PROGRAM

START TO END OF PROGRAM

The primary objective of Phase I of the above program (as outlined in the abstract of our proposal) has been to introduce an additional dimension of control to the process of making high temperature superconductors. To the best of our knowledge, this additional process variable has not been used by anyone in the field of high temperature superconductors. In its previous research, supported by the National Science Foundation, USACO has been able to achieve dramatic and unexpected results in triggering nucleation of desirable crystal structures common to ferrous, non-ferrous, and ceramic systems.

The initial approach taken by the USACO Phase I proposal has an inherent provision to ultimately achieve continuous production manufacture of high temperature superconductors.

As a result of a 14 year research and development effort, USACO has developed the first completely automated continuous manufacturing system to produce ferrous and non-ferrous wire. The initial family of products made from this super strength wire will be available in 1991. Many of the patentable features of this completely automated, continuous, system entitled the Thermastress Miniplant Technology (TMT), have been found to be applicable to the ultimately contemplated Continuous Production of high temperature superconductors.

Statement "A" per telecon Phoebus
Williams. U.S. Army Strategic Defense
Command/CSSD-AT-E. P.O. Box 1500.
Huntsville, AL 35807-3801.
VHG 11/26/90

The basic objectives of Phase I were limited to looking for discernable improvement in significant physical characteristics in a well known high temperature superconductor material - Yttrium Barium Copper and Oxygen (1,2,3) subjected to externally induced shear during the crystallization process.

Recognizing constraints imposed by the budget available for the Phase 1 effort, no attempt was to be made at this stage to solve the problem of inherent brittleness of ceramic superconductors in order to produce a conductor which could be wound in coils. This problem remained to be addressed in a subsequent program. Amongst approaches considered were bundles of filament size conductive elements and possible addition of benign additives such as silver.

An essential requirement of the Thermastress process is to properly shape the cooling curve (T°-t) of the material as the externally induced shear is simultaneously applied to this material. This is achieved by establishing a desired temperature gradient along the length of the material being processed. See Figure 0.1.

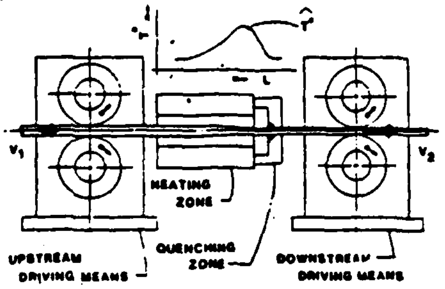

FIG-0.1

Monitoring this temperature in previous research programs was accomplished with an optical pyrometer taking direct readings from the specimen being processed. To obtain such temperature readings reliably this specimen diameter must be sufficiently large to allow all of the pyrometer target to be on the specimen. Consequently the approach required the specimen to be of substantial diameter. The decision was also made to maintain the specimen straight throughout the steps of their preparation, processing through the Thermastress Process and all subsequent handling.

To subject high temperature superconductor material of Yttrium-Barium-Copper & Oxygen to the Thermastress Process, the high temperature superconductor material was to be encapsulated between the copper wire core substrate and the outer cladding (See Fig. 0.2). Initially high temperature superconductor material was to be deposited by plasma.

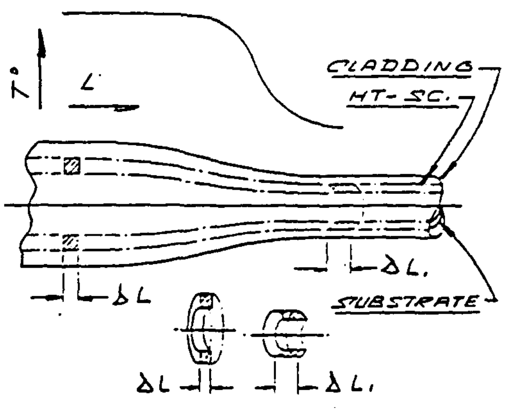

FIG-0.2

Fig. 0.2 Sectional view of encapsulated wire

For this purpose a simple apparatus was to be used to hold, rotate, and move the wire axially relative to the plasma gun.

The application of silver on the substrate material and the formation of the outside cladding material lent themselves to an electrodeposition process. A novel method was devised whereby the proposed apparatus was modified and adopted to both the deposition of high temperature superconductor material by pyrolysis and also to the electrodeposition of required layers of silver and copper cladding. (See Figure 1.) The use of copper wire as a substrate provided the means of accomplishing the successive separate steps on a continuous basis.

A patented annular orifice atomizing nozzle previously developed for shaping the Temperature Gradient in the Thermastress Process was adopted for the application of solution of Yttrium Barium and Copper oxides in an aqueous solution of nitric acid. The core wire held at both ends in collets was to be moved relative to the pyrolysis and electrodeposition means. Since the wire had to be held and supported over a span of approximately 10 feet (3 m) and had to pass concentrically through various annular nozzles and conforming electroplating anodes, a system of retractable supports was worked out and incorporated in the design of the system.

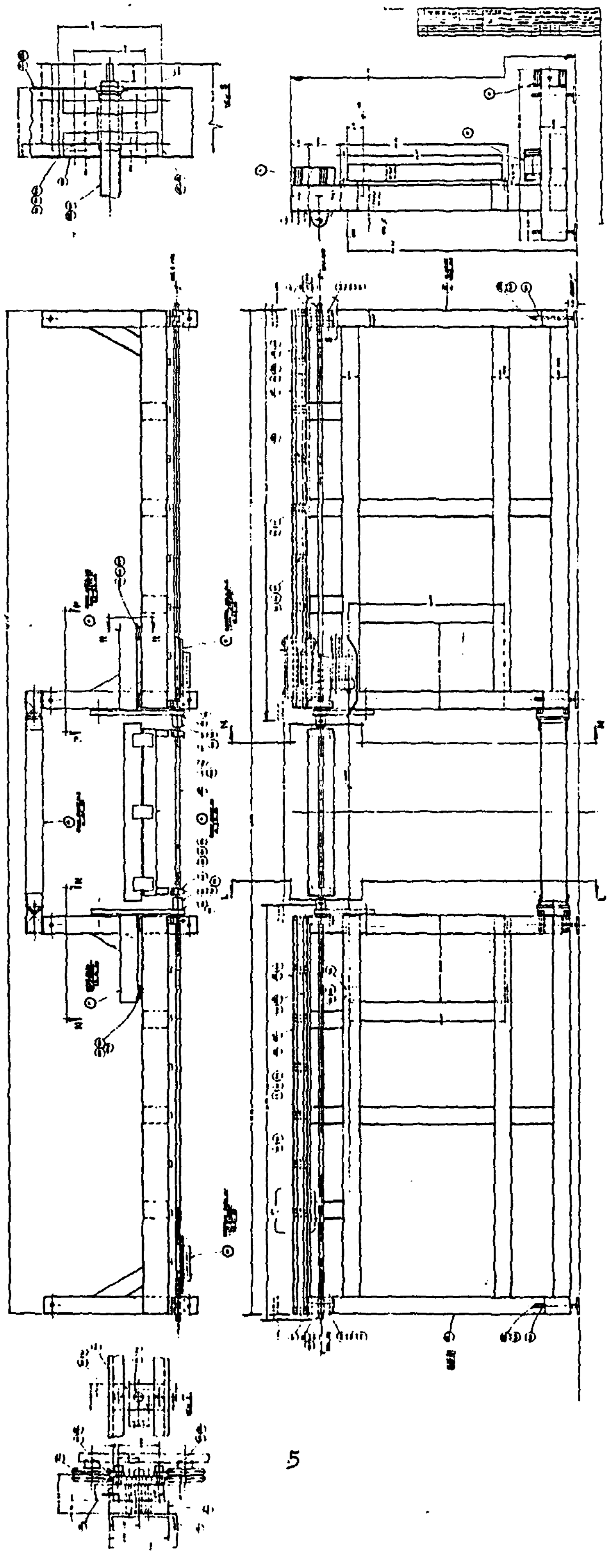

FIG-1

The means of moving the wire substrate had to be provided with a precise speed control covering a wide speed range in order to obtain the optimum deposition rate.

At this point in the program, the next logical step to the attainment of the program's basic objective was to make it possible for the two collets to move at independently controlled speeds V_1 and V_2. To provide for this, the drive system for the apparatus was modified further so as to enable control of V_1 and (V_2/V_1) independently. The magnitude of V_1 affects T°-t relationship. The ratio (V_2/V_1) determines the elongation imposed on the material processed and consequently the magnitude of induced shear within this material. By adding this feature it now became possible to carry out all the necessary work on the same apparatus without having to remove the specimen from the apparatus.

This eliminated subjecting the high temperature superconductor material to cracking due to handling or crushing in the driving wheels of the Thermastress Laboratory Prototype #3 or (P-3). In our Phase I proposal P-3 was to be used for this purpose. P3 was available from previous research programs.

As to be anticipated, the above changes substantially increased the size and degree of sophistication of the apparatus and brought its estimated cost commensurately above the amount available within the Phase I budget. However, additional information gleaned through research relating to martensite phase transformations in ceramics reinforced our conviction that the approach towards achievement of the project objective was homing in on the target.

Furthermore, when the program was well on its way, reports appeared in technical literature of successful application of a conveyor-type approach to continuously carry out several steps of the high temperature superconductor production process.

The wire core substrate, as initially proposed by USACO, constitutes, in essence (as intended from the beginning) continuously moving conveying means for the high temperature superconductor throughout the ultimate production process. By splicing on the fly additional wire with an automatic flash welder (as carried out in the USACO TMT ferrous and non-ferrous miniplants) it will make possible continuous uninterrupted production of a high temperature superconductor.

After due consideration a decision was made at USACO to proceed with the program to the conclusion of Phase I at no additional cost to the government. To finance the amount over and above monies expended from the Phase I budget we are currently actively seeking investment from outside sources. At present the Engineering and all working drawings for the above mentioned apparatus are complete and the major long delivery purchase items have been purchased and are now on our shop floor. See reduced engineering drawings (Figure 1 to Figure 12) attached.

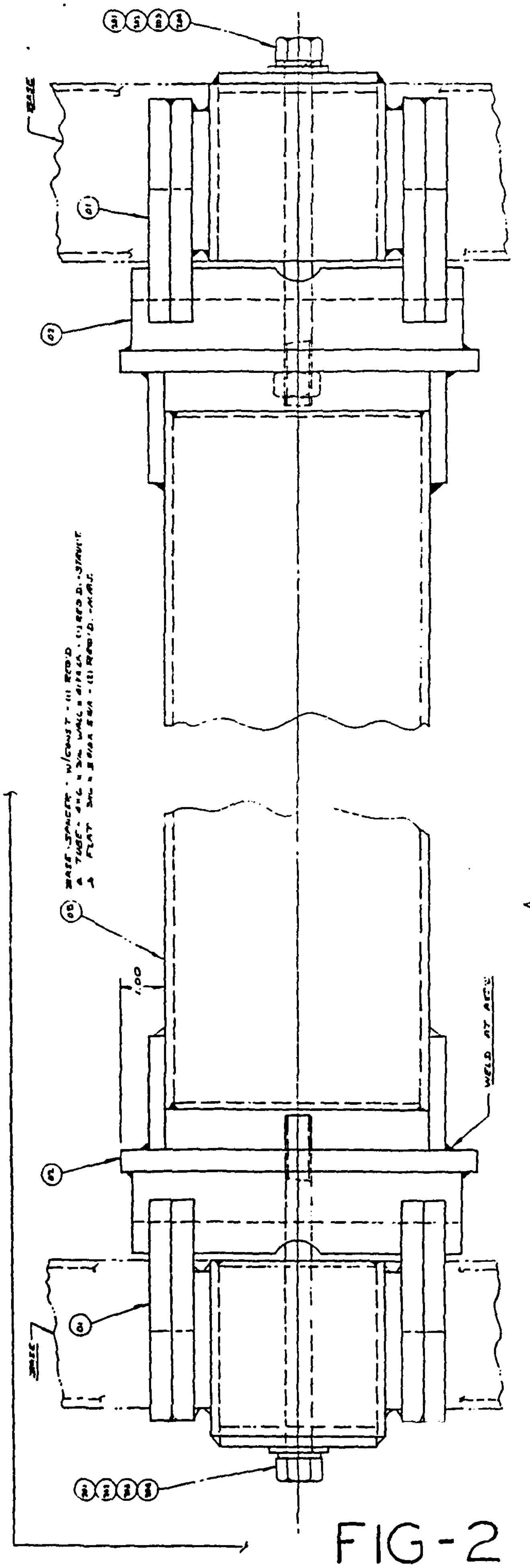

FIG-2

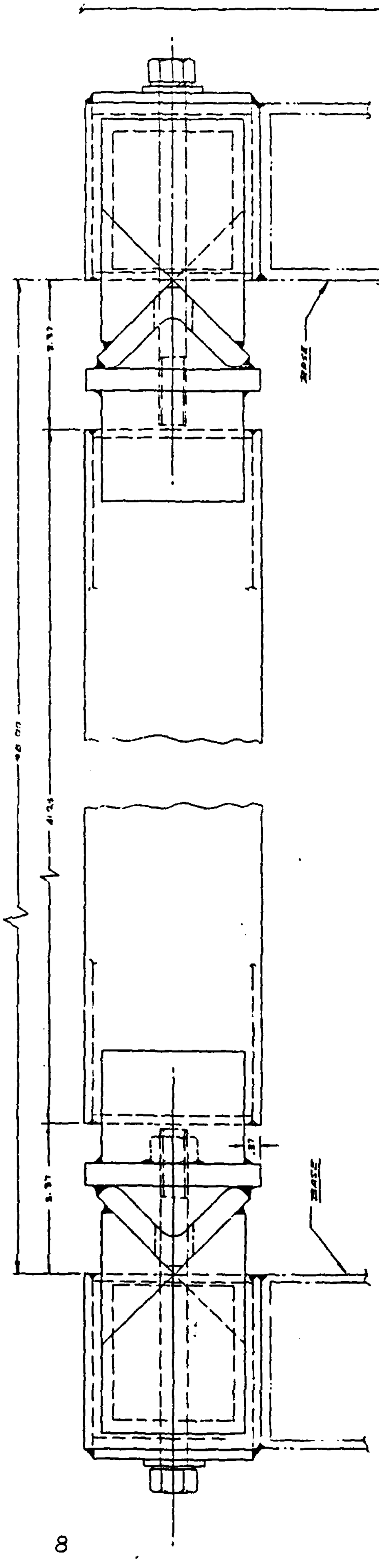

FIG-3

U.S. AUTOMATION CO.

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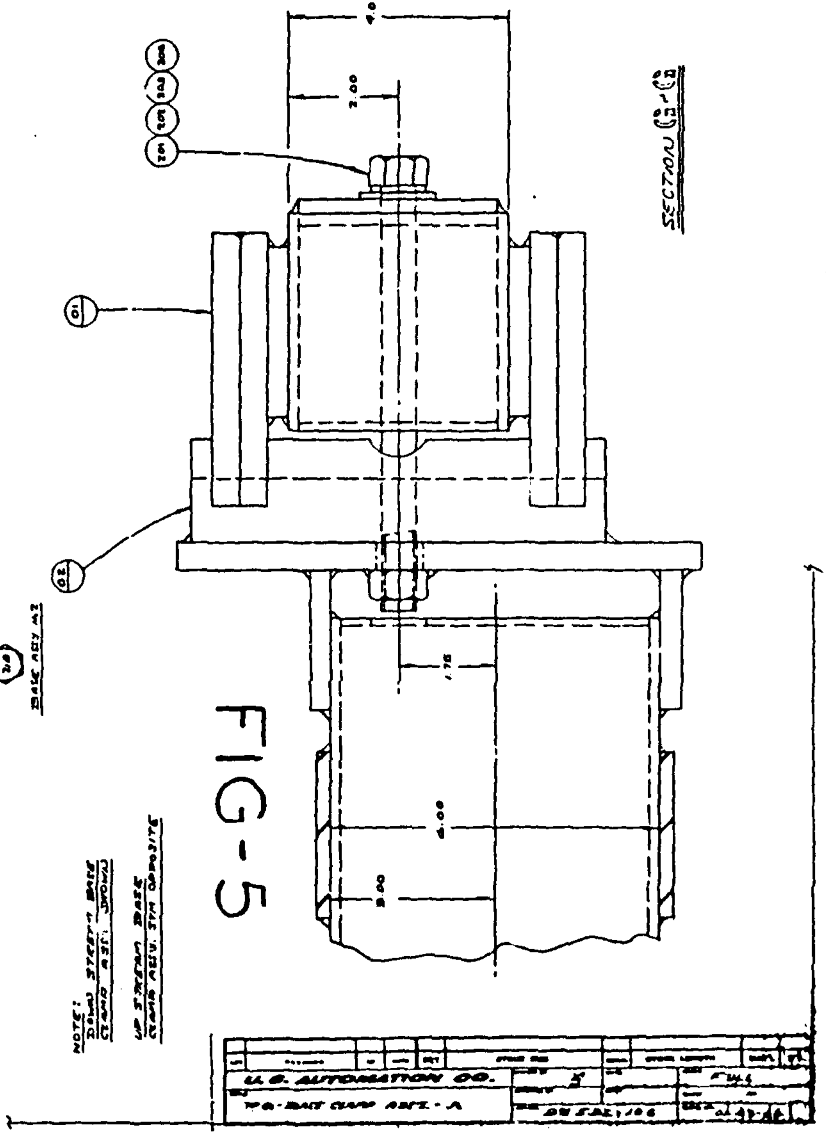

FIG-5

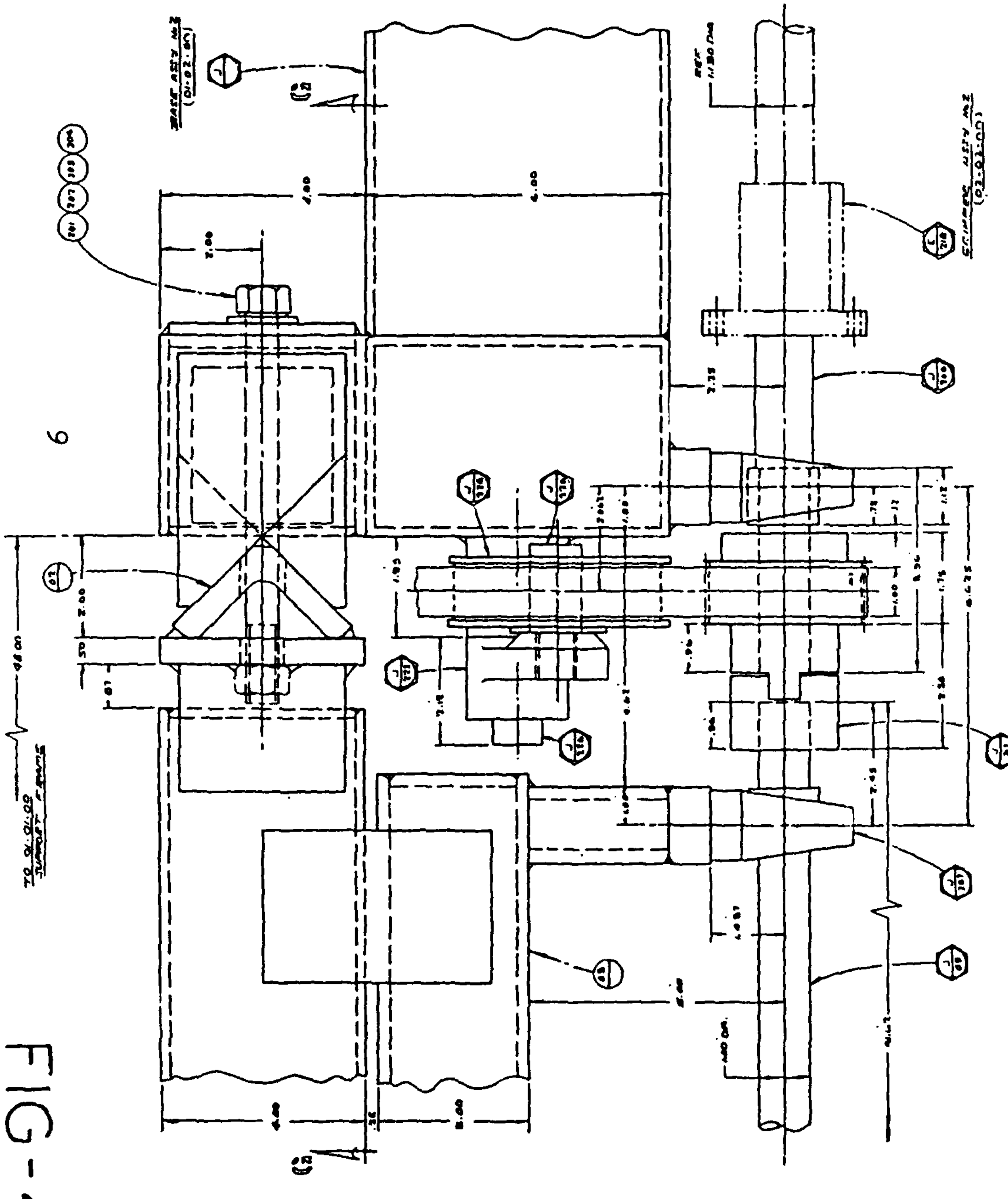

FIG-4

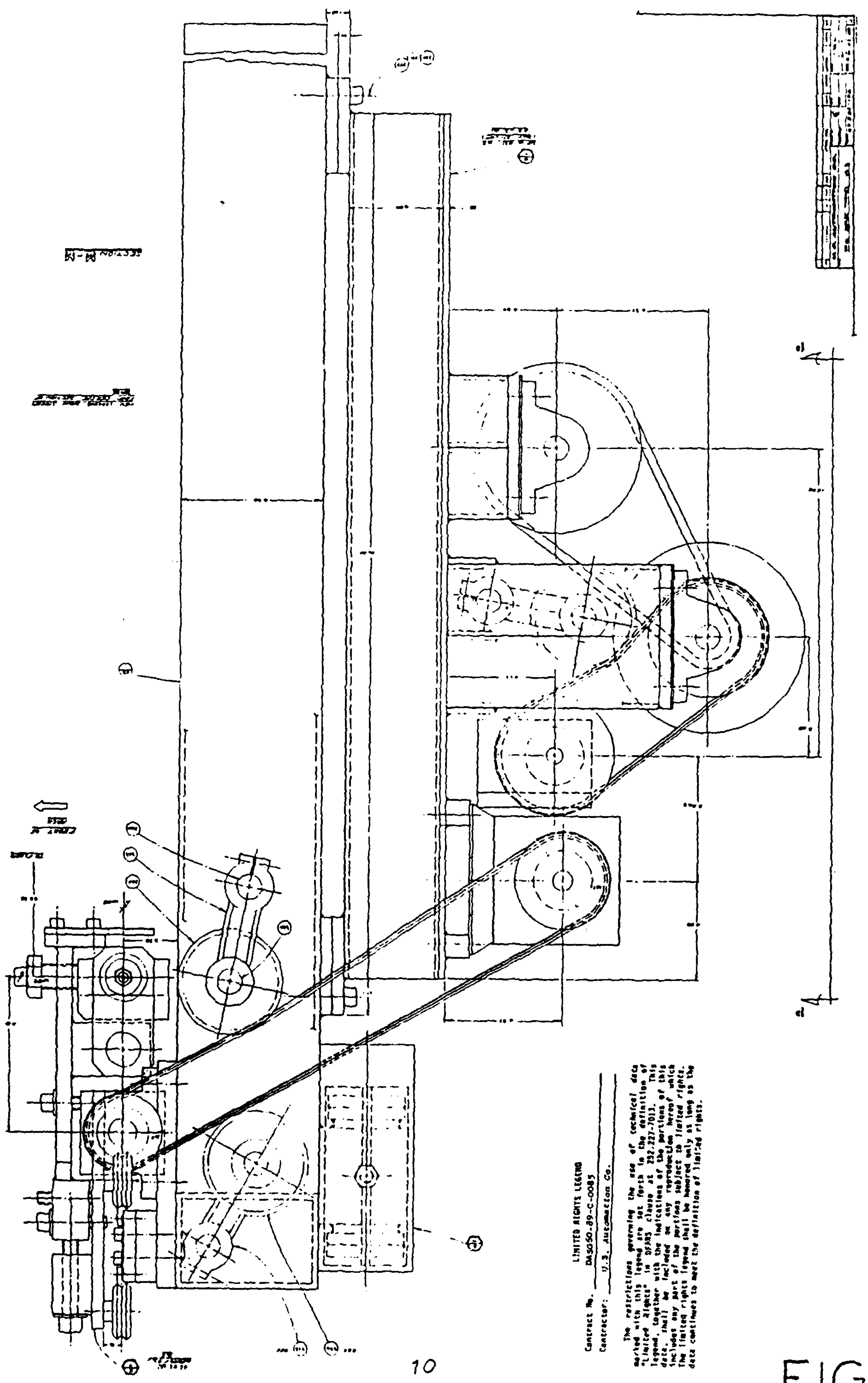

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FIG-6

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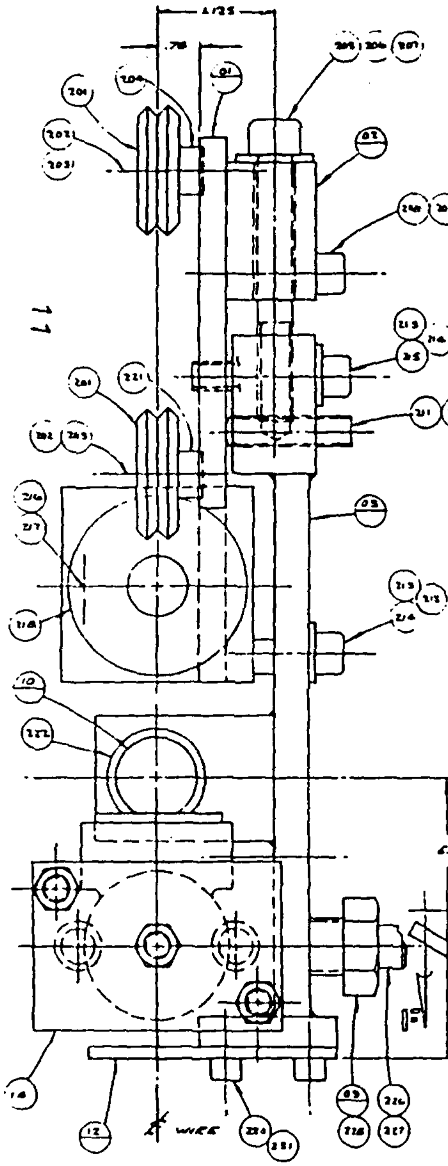

FIG-7

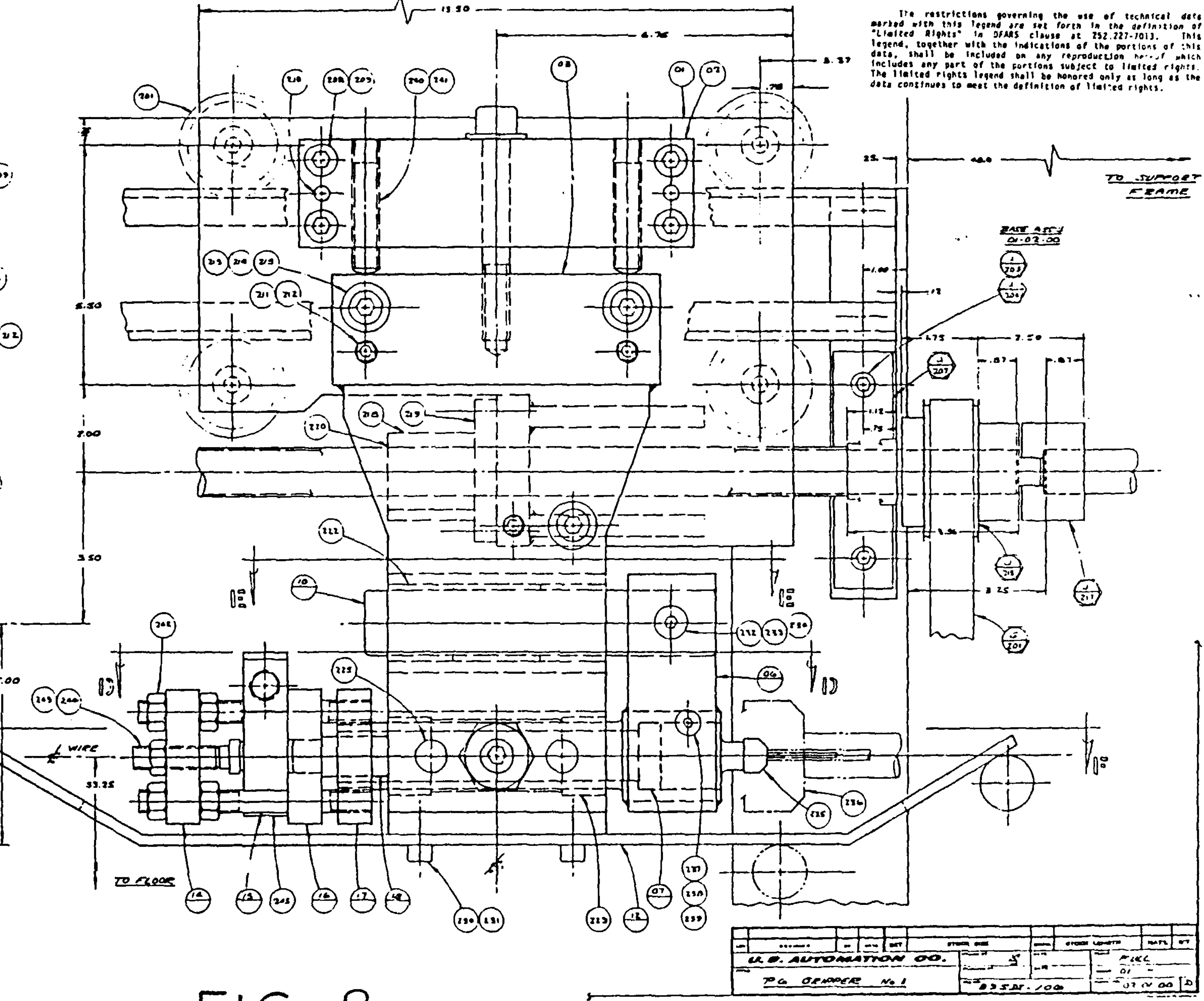

FIG-8

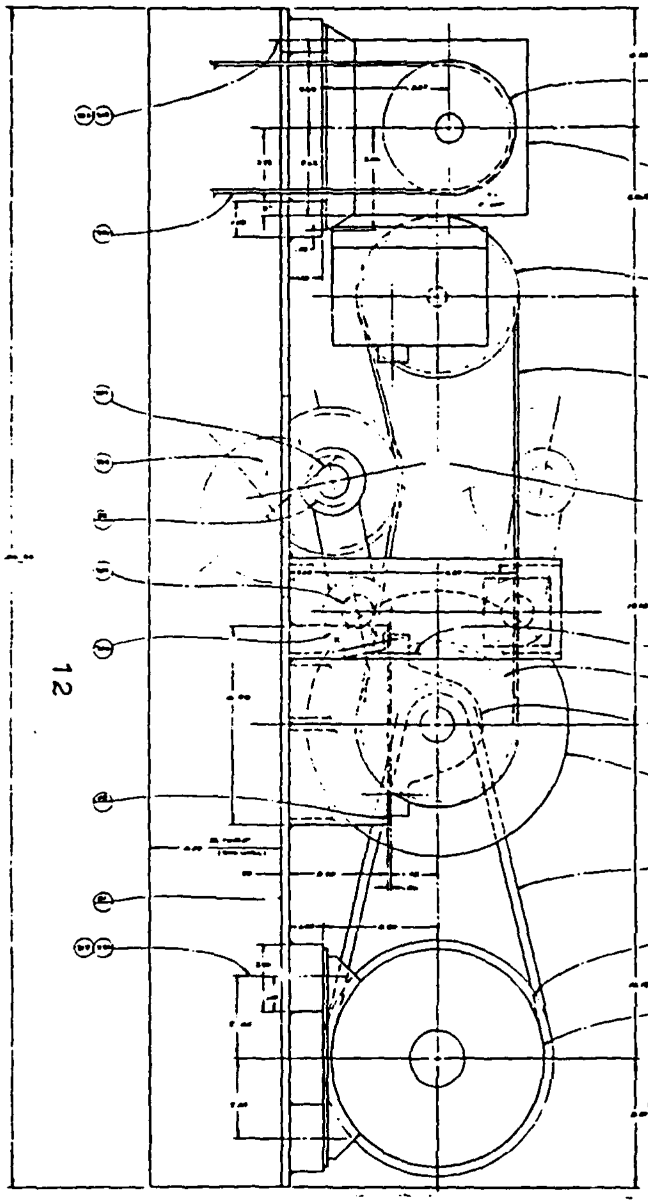

FIG-9

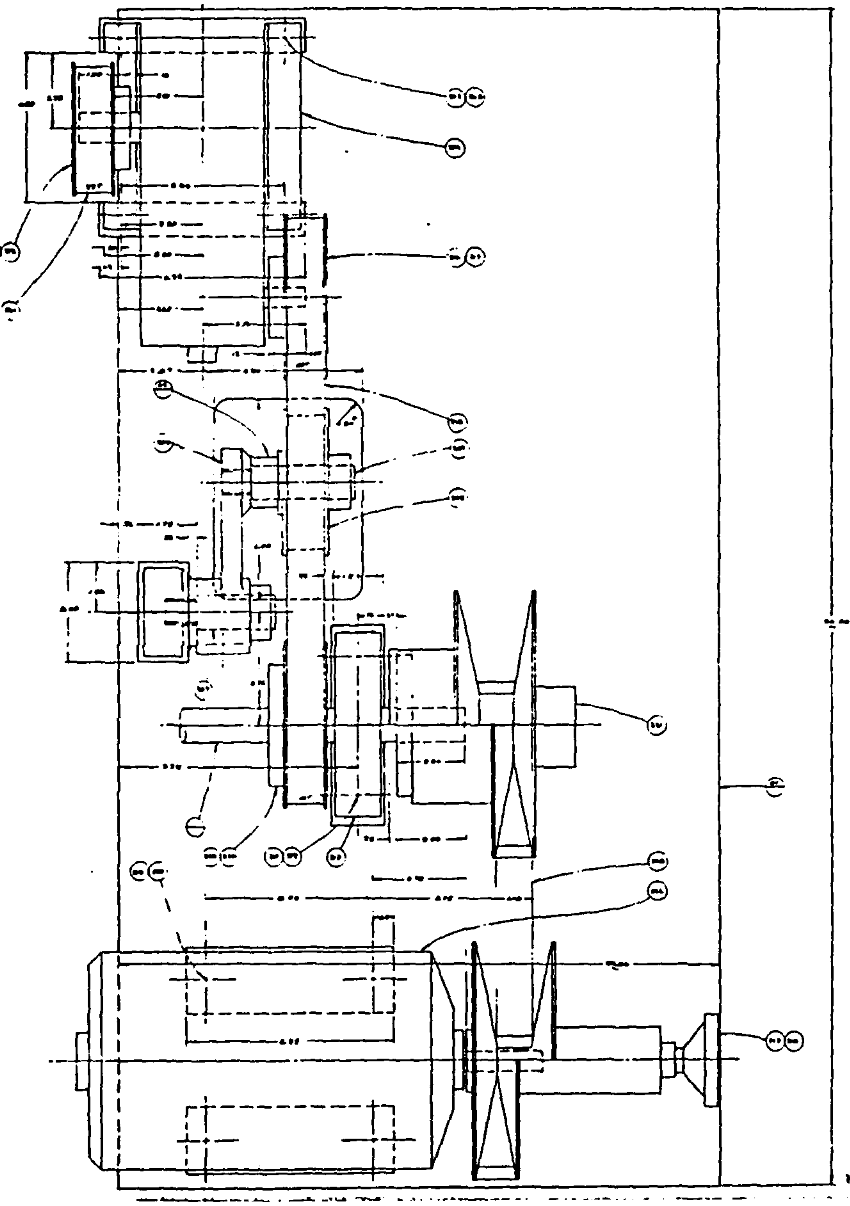

FIG-10

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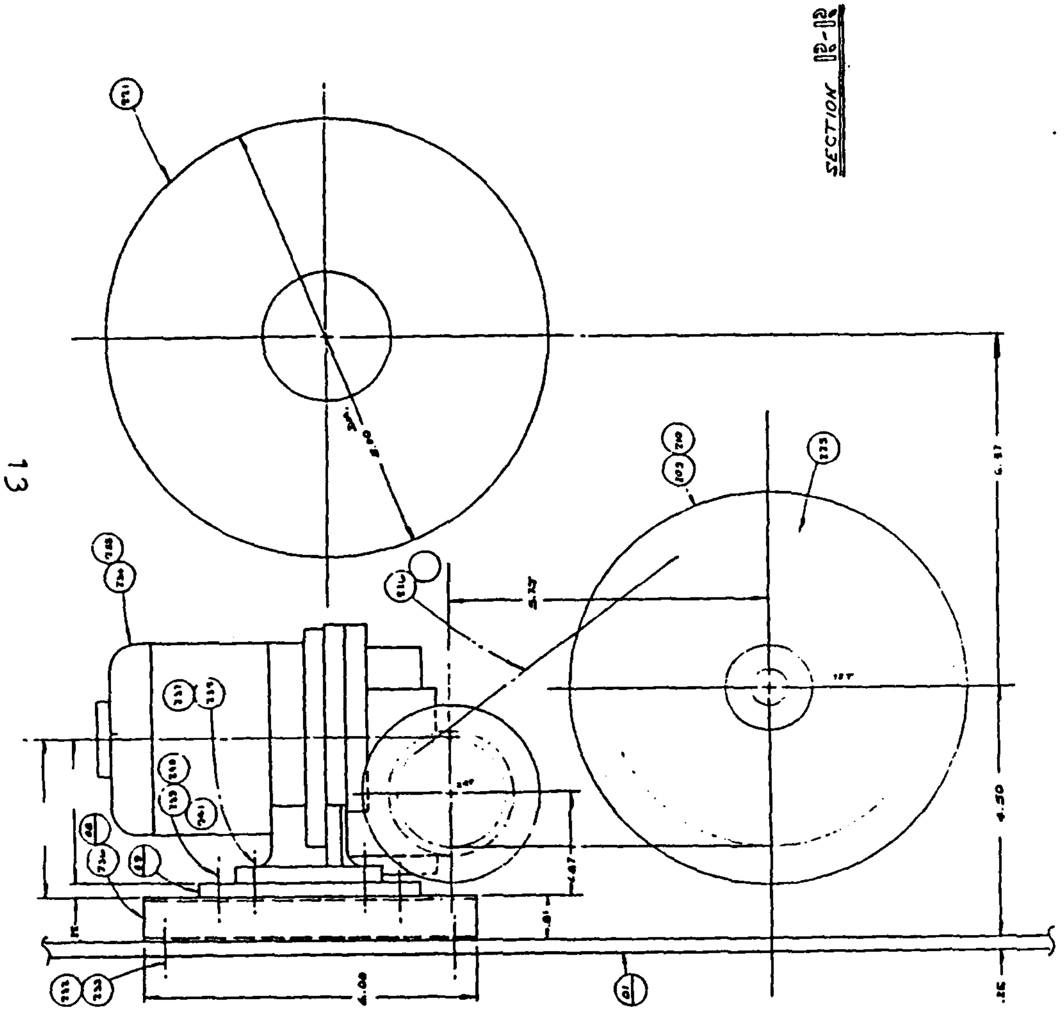

FIG-11

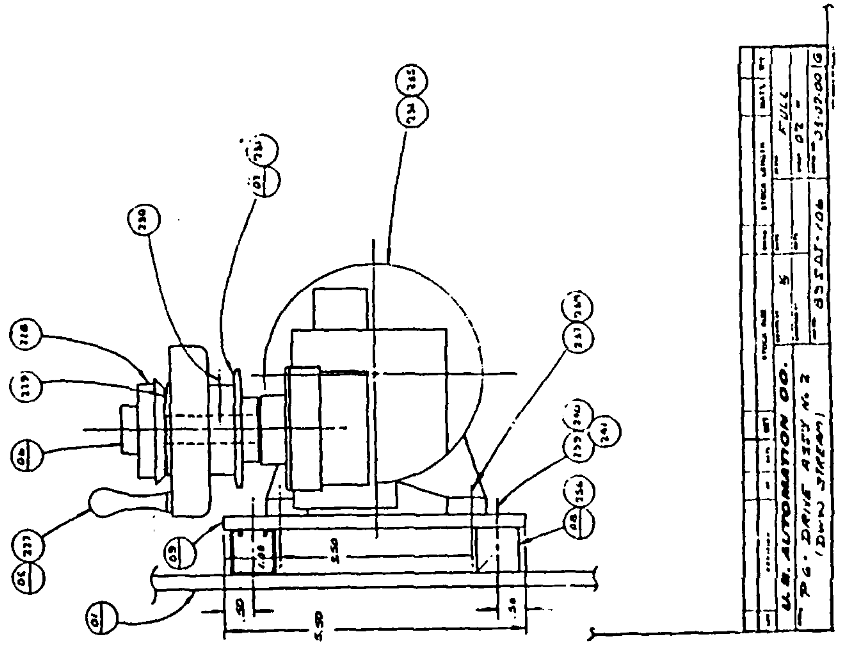

FIG-12

PHASE I REPORT SUMMARY

In the course of pursuing the primary objective of Phase I of this research effort, (as outlined in the original proposal), the method to achieve its attainment has been substantially refined and modified. This now greatly enhances the probability of achieving favorable results in attainment of this initial objective.

The progress made to date is a result of:

(i) Extensive on-going search for data linking martensitic transformation in ceramics and the formation of perovskites.

(ii) Examination of the developments in high temperature superconductor research worldwide as reported in various journals and publications. This was carried out, and continues to be carried out, with the specific objective of determining how these developments may negate or support the validity of our assumptions.

(iii) Additional novel ideas generated by the USACO R & D team in the course of seeking solutions to the remaining unsolved problems.

An integral part of this research is the effect of the Thermastress process developed at USACO over a period of approximately 14 years. The Thermastress process was initially developed to induce martensite and bainite transformations hitherto unobtainable in low carbon ferrous alloys.

Additional major steps towards the ultimate objective of creating a viable continuous manufacturing system have been covered on a theoretical as well as apparatus design level. The findings of the Phase I effort now offer an **OPPORTUNITY** to bridge the gap between laboratory research phase and a viable high temperature superconductor continuous production process pilot line.

The feasibility of this new promising objective can be established by a testing program to be carried out on the previously developed and engineered apparatus with the addition of necessary modules (as shown in attached reduced working drawings Figure 1 to 12 above) to incorporate pulsating tensile force.

The entire contemplated continuous production process, to be attained ultimately, can be tried out (and developed) on this new Continuous Production Feasibility Module (CPFM) on a station by station basis.

TITLE PAGE

1. Pulsating Tension
2. Proposed HT-SC Conductor Configuration
3. HT-SC Proposed Conductor Ribbon Element Configuration
4. Brief outline of Proposed Phase II Program
   Continuous Production Feasibility Module (CPFM)

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PULSATING TENSION

In USACO's Thermastress U.S. patents, issued to date, the added process variable to conventional recrystallization kinetics (dependent on the nature of the material and the shape of its cooling curve alone) is simultaneously applied deformation. This deformation is achieved by stretching the material as it is moved through immediately adjacent heating and cooling zones. It is a well known fact that whether a specimen is subjected to external axial tensile or compressive stresses, an important internal end result is the same. Namely, that a shearing strain occurs along planes at an approximate 45 degree angle to the axis along which the external forces are applied. We consequently have identified the added process variable - (deformation) present in the USACO Thermastress process as externally induced shear.

In the Thermastress process, as it has been applied to date, the externally induced shear takes place within the Reduction Cone of the material. See Fig. 1). This Reduction Cone is normally of relatively short length. This limitation is primarily due to the fact that as the length of the Reduction Cone increases, and the included angle within the cone approaches zero, it becomes progressively more difficult to prevent irregular necking of the material in the reduction zone. The consequence of this is inconsistent cross section in the end product and loss of the dynamic equilibrium essential for stable operation.

In certain applications of the Thermastress process it is desirable to extend the period of time during which the material is subjected to externally induced shear stresses. In particular, information available regarding time required for various heating, cooling, and annealing steps required by previous methods of making high temperature superconductors
dictates the need to provide the apparatus with the capability of substantially extending the time period during which the specimen is subjected to externally induced shear.

A logical deduction from the preceding statements follow that if one were to impose axially a reversible force on the specimen (which is moving through the Thermastress process) then one will achieve induced shear within the specimen over its length (which is confined between the upstream and downstream driving means).

To prevent buckling of the processed specimen during the compression portion of the pulse cycle a minimal amount of tension is maintained during the reversal of the pull from its maximum to its minimum values (See Figure 2). Hence the resultant of this is a pulsating tensile load applied to the processed work simultaneously, as it is passing through the appropriately controlled temperature zones. In order to prevent fracture of the ceramic material, the magnitude of maximum elongation or the maximum corresponding force imposed on the specimen must be below 0.4%.

The pulsating tensile stress will be applied on the material being processed, thereby inducing a pulsating internal shear on the entire length of material confined between the two driving means. Means will be provided to adjust the frequency of vibration to attain the best frequency suited for a given specimen. It will also be possible to select the desired minimum tensile preload. The magnitude of the maximum pulse force will be adjustable with calibrated means. This enables extending the period of time the material is subjected to pulsating shear manifold without reducing the rate at which the end product is exiting from the line.

The advantage of externally applying pulsating tension is to enable the material to be subjected to simultaneous action of externally induced shear while it is going through the various temperature zones. The added benefit is to substantially reduce the severity of the deformation and enable a very much gentler shear strain (δ) to be imposed on the material. This is particularly pertinent to high temperature superconductors which are inherently brittle.

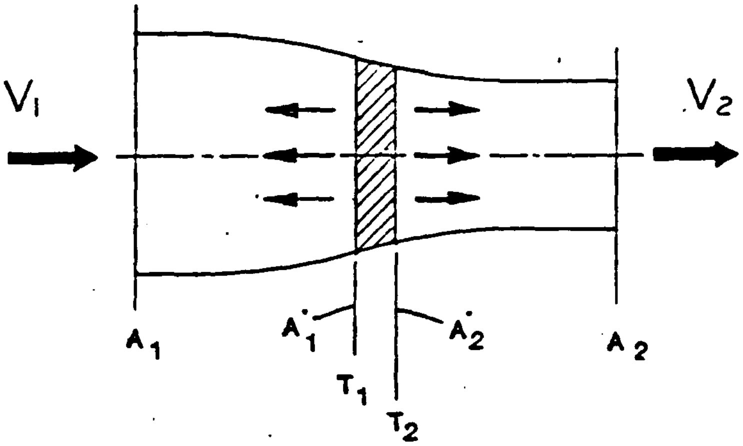

FIG-1

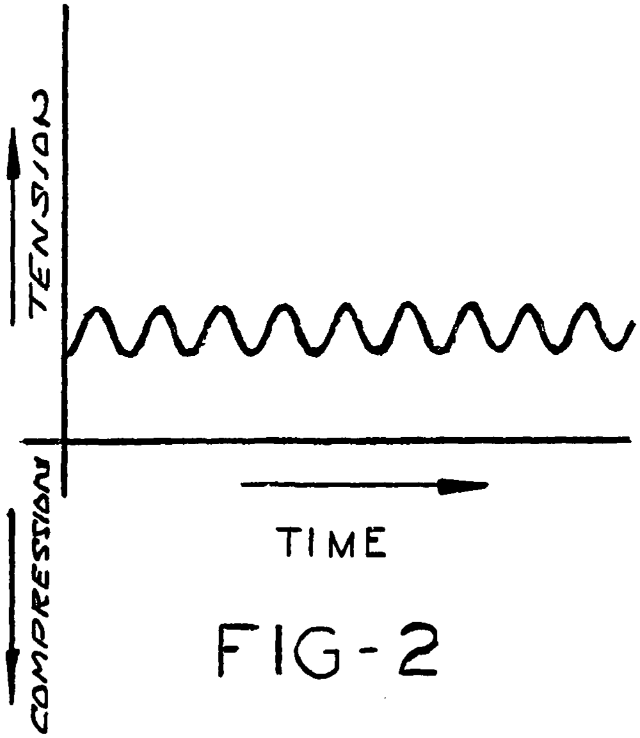

FIG-2

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PROPOSED HT-SC FLEXIBLE CABLE CONFIGURATION

Two major obstacles in the way of utilizing ceramic high temperature superconductors in existing applications have been:

(1) Their brittleness has prevented winding the high temperature superconductors into coils within a practical range of diameters.

(2) The inability of the HT-SC's to carry a substantial current per square centimeter (J_c) has limited practical applications.

To address these problems, U.S. Automation proposes that a flexible HT-SC cable be made of a stack of superconductive ribbons free to slide relative to each other within an insulating sheath (See Fig. 1). This insulating cover could be wrapped around a bundle of superconductive ribbons in a conventional cross basket weave manner. Or it could be extruded through a nozzle to form a plastic casing around the conductor bundle as it is moving continuously through the manufacturing process.

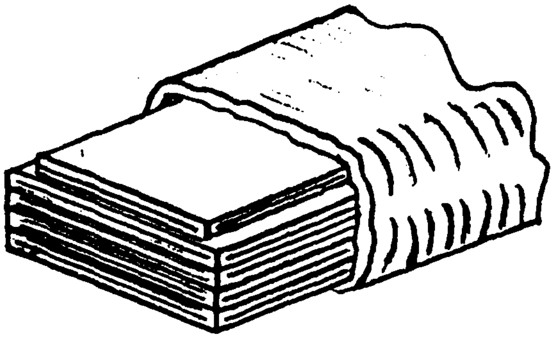

FIG - 1

On the basis of the previous numbers derived from a report published by General Atomics, Inc., a ribbon, such as proposed in our program, would have a total thickness of 0.010 inch (254 μm) and would be capable of being wound around a diameter of about 1.66 inches. A smaller diameter could be made possible with a proportionally thinner ribbon.

The addition of silver will further improve the ability of the superconductive ribbon to be bent around a practical radius to be used in useful applications. It's purpose is also to <u>minimize</u> the possibility of weak links and cracks in individual layers of material.

The effect of stacking separate layers of high temperature superconductors, interspersed with thin films of silver, should also tend to <u>contain</u> any weak links or fractures occurring in the individual layers within those individual layers.

The basic advantage of the proposed approach, from the manufacturing standpoint is, that, once the problem of retaining superconductivity in each preceding layer is overcome, it will be possible to build up the cross section of conductor elements simultaneously (See Figure 2).

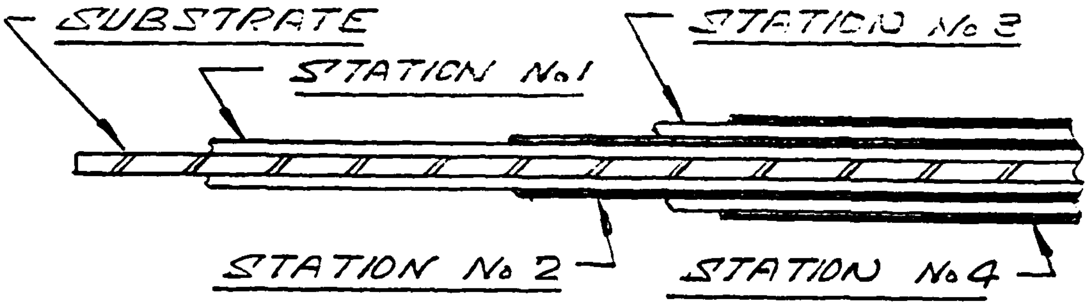

FIG- 2

In other words, as the core substrate of the superconductive ribbon is moving through the system, all the layers are being deposited one on top of each other simultaneously starting with the first layer in station 1.

In this manner, doubling of the production rate can be accomplished by doubling of the number of stations.

The proposed basic approach as applied to the continuous production pilot plant envisions extensive utilization of the various drives and mechanisms which have already been developed for the manufacture of superstrength steel wire in the novel Thermastress Miniplant Technology (TMT) approach. The necessary drives to propel the ribbon through the system from station to station have already been developed and are capable of carrying out the necessary functions as outlined in the proposed approach. The approach of building up a conductor from ribbon elements leads to simple effective friction drives to propel the work through the process.

However, in the next immediate phase of the program a modified version of the P6 laboratory prototype called Continuous Production Feasibility Module (CPFM) will be necessary. It is essential because it specifically provides for all the necessary process variables which have to be provided at various stations of the proposed manufacturing line. Within this one test cell it should be possible to duplicate what has to be accomplished in the complete manufacturing line. Passing the same specimen through the various steps will correspond to the various stations within the manufacturing line.

The entire USACO approach works back from the concept of a complete manufacturing line towards individual processing stations with the objective of developing them such that they can be integrated into one continuous manufacturing system. U.S. Automation Company has 15 years of experience in solving problems of this nature in the development of the first continuous manufacturing process for the manufacture of metal wire. The initial wire produced by U.S. Automation Company was for nonconductive purposes but it was enhanced with outstanding superstrength characteristics accomplished by the patented thermomechanical process developed by USACO. Consequently, U.S. Automation has brought to bear on this very interesting problem its expertise both in the area of computer controlled continuous automation processes and also its expertise in the area of modifying microstructures of materials on a continuous manufacturing process basis.

HT-SC PROPOSED CONDUCTOR RIBBON ELEMENT CONFIGURATION

Each individual conductor ribbon element will comprise of a thin (≈0.001 inch) core substrate with a build up of several high temperature superconductor films interleaved with layers of silver. Material produced by a Rapid Solidification process is presently under consideration for use as the core substrate.

This core ribbon will first have a thin film of silver (Ag) deposited on it by electrodeposition. A layer of high temperature superconductor will then be deposited by pyrolysis process onto the Ag layer. This film would be continuously moved through a combustion chamber which will have appropriate temperature zones to enable the proper crystallization kinetics to take place and to form the desired high temperature superconductor crystalline microstructure.

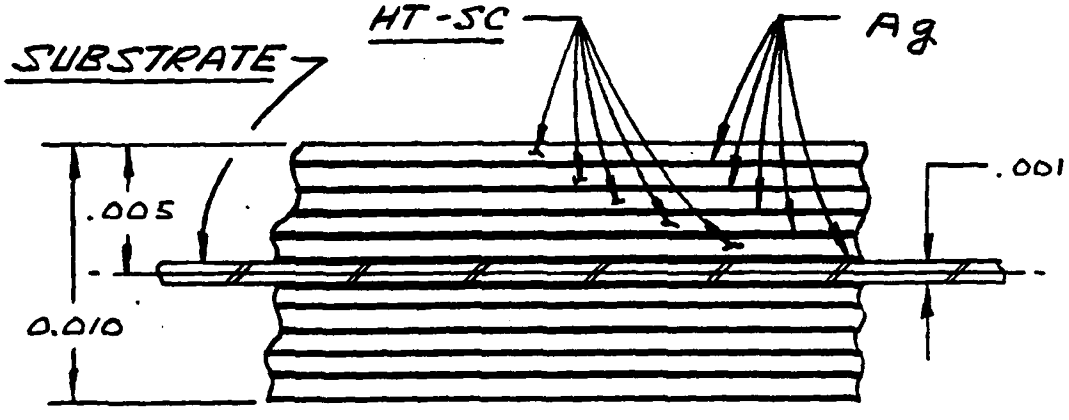

FIG - 1

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In a station immediately following pyrolysis, annealing (infusion of oxygen atoms into the previously deposited layer of high temperature superconductor) will take place. To provide the required oxygen a flame high in oxygen content, (lean flame) will be used in the special patented annular orifice burner previously developed for continuous operation on other Thermastress applications.

Subsequent to the deposition of HT-SC film, a thin layer of electroless silver will be deposited in a suitably designed tank with weir overflow. This Ag layer will provide a conductive surface at ambient temperature on the superconductive film. This operation will be followed by a layer of silver deposited by conventional electrochemical means. The process will be repeated in subsequent stations by depositing the HT-SC film followed by a film of electroless silver and a film of electrolytically deposited silver. The ribbon material will proceed from the stations within which this have been accomplished through the next series of stations where the process will be repeated. In this manner, layer by layer, superconductive film will be built up on both sides of the ribbon matrix until the desired cross section is achieved.

This cross section will be determined primarily by the radius of curvature that has to be used in the application of the material.

It is a known fact that ceramics obey Hooke's Law and their Modulus of Elasticity falls generally between 6 and 10 x 10^6. Hence they will deform on a linear basis up to the point of fracture which of course occurs at a much lower level of elongation than with steel.

With reference to this General Atomic Inc's research which was reported in High T_c News October 15, 1990 issue Volume 4 No. 20 we have the following important information.

Continuous superconductor wire was produced using rare earth, barium, and copper oxide. It comprised of 0.3 m diameter (d) filaments imbedded in a solid copper matrix 0.9 mm thick.
It was claimed that it was capable of being bent into a 15 cm diameter coil. From this we can arrive at the elongation at the outer fiber as follows:

<u>Length of arc at the neutral axis</u> = $\theta \cdot 2 \cdot \pi \cdot R/360°$

<u>Length of the arc at the outer fiber</u> = $\theta \cdot 2 \cdot \pi \cdot (R+r)/360°$

$E = -1 + (R + r)/R$

Diameter (D) = 15 cm, then R = 7.5 cm and r from the report is approximately 0.3 mm.

Therefore $E = [(75\ \text{mm} + 0.3\ \text{mm})/75\ \text{mm}] - 1 = 0.004$

This translates into 0.4% elongation.

This means that it was possible to stretch this HT-SC 0.4% without fracturing it. Note that with steel the maximum E at rupture is approximately 20%. Addition of silver is intended to provide a lubricating film between layers of superconductor as well as to provide silver atoms to migrate through the HT-SC film itself. These measures will provide an improved ability of the SC material to withstand bending and to inhibit weakening or fractures. By limiting the total thickness of a conductor element ribbon to 0.010 inch (254 μm) it should be possible to wind it around a 1.00 inch (25.4 mm) diameter circle.

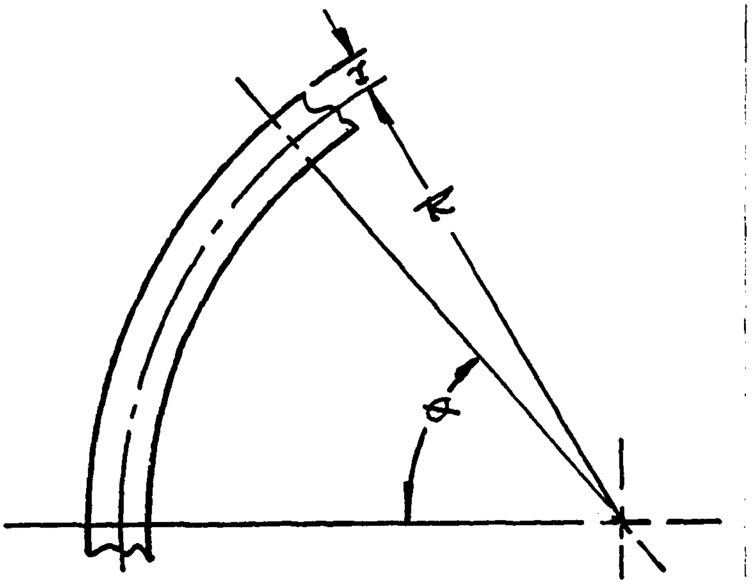

FIG-2

BRIEF OUTLINE OF

PROPOSED PHASE II PROGRAM

CONTINUOUS PRODUCTION FEASIBILITY MODULE (CPFM)

The basic approach to investigate the effect of externally induced shear on formation of high temperature superconductor crystals, proposed now, is outlined as follows:
A thin (0.025 mm by 2 mm wide) metallic ribbon will be used as a substrate on which to build up the optimum thickness of superconductor element. Nickel ribbon produced by Rapid Solidification process is presently a favored candidate for the substrate material.

This width of ribbon will enable the optical pyrometer to monitor by continuously scanning the temperature gradient along the length of substrate between driving means. Continuous ribbon as a core substrate provides conveying means making it possible to ultimately achieve continuous manufacture of high temperature superconductors. To accomplish this end objective, each station in the production process will be designed with continuous movement of the material in mind. The primary function of the Continuous Production Feasibility Module (CPFM) is to determine the optimum values of the process variables as required within <u>each separate step of the manufacturing process</u>.

The main factor, which will add a <u>new dimension of control</u> to the process, is externally induced shear within the material as it moves through its crystallization kinetics cycle. This will be accomplished by subjecting the length of ribbon on which the high temperature superconductor material is being deposited to stretching as it is moving between two driving means. A pulsating tensile force will be superimposed on the section being processed.

The sum of the forces will be such that the material will be subjected to a pulsating tensile force the strain from which will be below that at which fracture occurs.

Control of (V_1/V_2), the ratio of the incoming velocity (V_1) and outgoing velocity (V_2) of the section which is subjected to externally induced shear.

In the formation of most crystals, and especially during the martensitic phase transformation present in ferrous, non-ferrous as well as ceramic materials, the shape of the cooling curve is of vital importance. In order to shape an ideal cooling curve as well as an ideal heating curve, CPFM will be provided with zones within which the temperature will be independently controlled. This applies both to providing controlled heat input means as well as, when necessary, providing means to remove heat from processed material as it is advancing through the CPFM. This principle has been patented in its application to manufacture of wire (U.S. Patent 4,872,923).

The values of temperature along the material being processed will be monitored by an optical pyrometer which will continuously scan the length of the work processed between the driving means. A special gaging apparatus has been designed, built, and used previously (see Fig. 1) whose function is to determine the shape of the longitudinal profile of the material subjected to deformation. Figure 2 depicts schematically a section of material subjected to deformation which is referred to as the Reduction Cone.

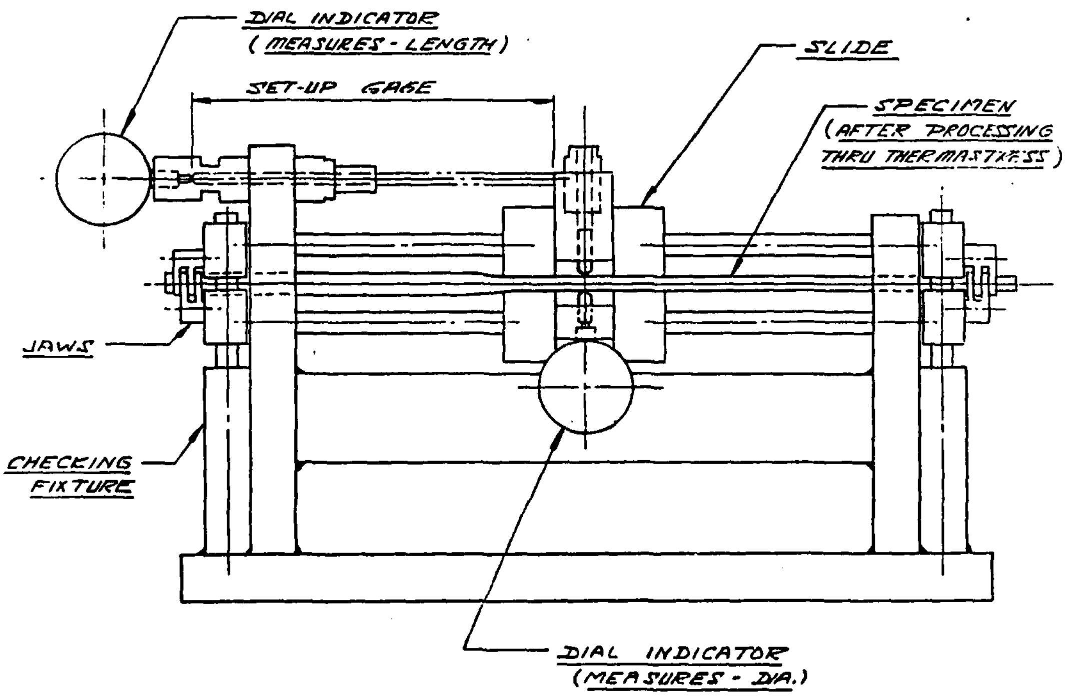

FIG-1

In Figure 1, V_1 is the velocity of material entering the Reduction Cone and V_2 is the velocity of work exiting the Cone. V_2 is greater than V_1.

Because $A_1V_1 = A_2V_2$

$$V_2 = A_1V_1/A_2$$

Consequently it is possible to determine velocity at any point along the length L of the specimen. From the above distribution of the temperature along the reduction cone (or its temperature gradient) it is possible to arrive at the temperature-time curve.

The above method assumes that a permanent elongation can be obtained without fracture as a result of the externally induced shear. In the event that in attaining a permanent elongation at the process temperature it will not be possible to prevent fracture of the high temperature superconductor material an alternate approach (B) will be investigated. This approach (B) employs application of pulsating external axial tensile force of a maximum magnitude below the level which will cause the material to fail. From previous analysis of the General Atomics data this should correspond to a stress required to produce 0.6% elongation. From available tables it appears that ceramics also have an elastic Modulus (from 6×10^6 to 10×10^6).

If elongation of this magnitude will not initiate fractures in high temperature superconductors at ambient temperature, as the findings of General Atomics, Inc. indicate, it is reasonable to assume that it should be less difficult to achieve it at elevated temperature.

The main difference between the two approaches A and B is that in approach B the stress imposed will be below the yield point of the material.

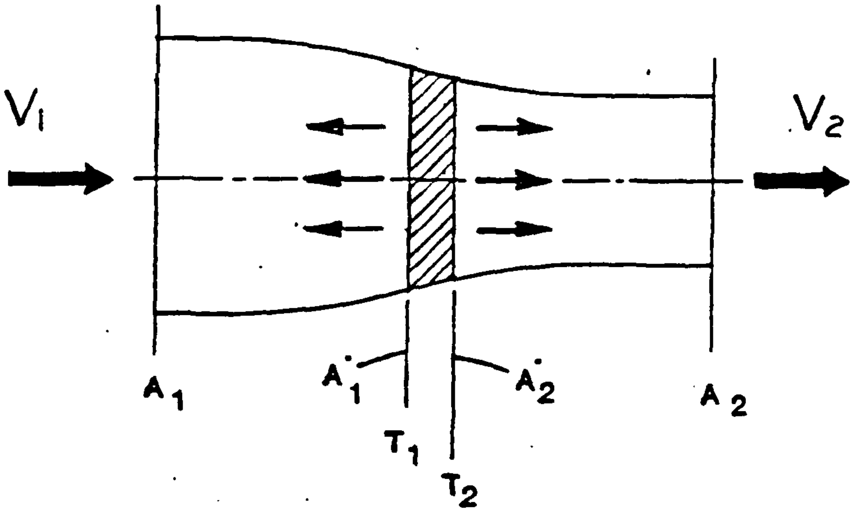

FIG - 2