

FABRICATION AND TESTING OF THE METAL HYDRIDE SORBENT BED ASSEMBLY FOR A PJLR10DJC 10 K SORPTION CRYOCOOLER

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ABSTRACT

The Brilliant Eyes Tern-Kelvin Sorption Cryocooler Experiment (BETSCE) is being developed for a future space shuttle flight to demonstrate 10 K sorption cooler technology in a microgravity environment. Three hydride beds circulate hydrogen through the various operation modes of the 10 K periodic cryocooler. Two beds, a fast absorption bed filled with $\text{LaNi}_4.8\text{Sn}_{0.2}$ alloy and a low pressure bed with ZrNi as the sorbent material, sequentially absorb hydrogen to drop the pressure from 0.25 MPa to below 0.2 kPa within two minutes. The low pressure bed also maintains the refrigerant pressure at <0.2 kPa as solid hydrogen sublimates with $T < 11\text{K}$. The third bed, which is filled with the $\text{LaNi}_4.8\text{Sn}_{0.2}$ alloy, collects the hydrogen from the other beds at nominal 0.1-0.15 MPa in order to compress it to 10 MPa for transfer to a storage reservoir. Each bed incorporates design features that enhance heat transfer and hydrogen gas flow in order to meet performance goals. The three hydride beds and the complete sorbent bed assembly (SBA) have been built and the beds were tested in the laboratory. During measurements under various reaction conditions including those that simulate flight operation, the hydride beds were found to have reversible hydrogen storage capacities that exceed the BETSCE system requirements. The hydrogen absorption kinetics permit the absorption of appropriate quantities of hydrogen to meet the cool down goal of 10 K within 2 minutes. The completed SBA has been integrated with the other components of the BETSCE cryogenic refrigerator for system ground testing.

INTRODUCTION

Sorption cryocooler technology provides opportunities for reliable, low vibration, and long-life cryogenic cooling of spacecraft borne infrared sensors. In those applications which involve intermittent operation at the low temperatures, the rapid cool down capability and low average power consumption of a closed-cycle periodic sorption cryocooler is especially attractive. Johnson and Jones [1] recently proposed a novel sorption cryocooler concept to generate quick periodic cooling from nominal 65 K to 10 K via the sequential formation of liquid and solid hydrogen by the Joule-Thomson (J-T) expansion of hydrogen gas. In a crucial verification that a ZrNi hydride sorption pump can be used to solidify and cool the condensed hydrogen refrigerant, Wu, et al. [2] achieved temperatures below 10 K in less than 2 minutes and maintained simulated sensor heat loads of 150 mW below 11 K for over 30 minutes. In order to advance the periodic 10 K sorption concept towards applicability in surveillance satellites missions, the Brilliant Eyes Ten-Kelvin Sorption Cryocooler Experiment (BETSCE) program was undertaken to design and build a demonstration system for earth orbiting operation. The objectives and key technology issues that are addressed by the BETSCE project were described in depth at the 7th International Cryocooler Conference [3,4].

A critical component of the BETSCE cryocooler is the sorbent bed assembly (SBA) which consists of three independent metal hydride beds that are mounted on a support baseplate. A separate radiator is connected to each bed to reject heat released during hydrogen absorption and as the beds cool after the desorption step. Wade, et al. [5] have thoroughly described the desired SBA performance goals and the various design features that have been incorporated to enhance hydrogen gas flow and thermal transfer within the sorbent beds. The present paper covers the fabrication of the SBA hardware and reports the results obtained during laboratory tests on the individual sorbent beds. It was found that the hydrogen absorption capacities and reaction rates for each bed exceeded the requirements that had been originally specified [3,4] for the BETSCE cryocooler.

SBA DESCRIPTION AND FABRICATION

The BETSCE sorbent bed assembly includes a fast absorption bed and a high pressure bed that both contain $\text{LaNi}_{4.8}\text{Sn}_{0.2}$ alloy powder as the hydrogen sorbent material and a low pressure bed with ZrNi as the sorbent. As shown in Figure 1, the SBA has the three hydride beds mounted on a baseplate along with an individual radiator panel, a manual isolation valve, and a pressure transducer on each bed. Supply lines from the SBA are connected [3,4] to the J-T cryostat assembly via a tank and valve assembly. A total of 8 type E thermocouples and 14 platinum resistance thermometers are attached to the three beds and their radiators. As discussed previously [5], the design of each bed was driven by not only the achievement of numerous hydrogen absorption and desorption performance goals but with a simultaneous satisfaction of "two fault-tolerant" space shuttle safety requirements. In particular, all vessel wall thicknesses were selected for 34 MPa proof pressures and hydride powder containment was addressed to avoid possible compaction and potential stress generation on the container walls as the hydride phases expand with hydrogen absorption [5]. Since the design details as well as the underlying rationale for their selection have been reported elsewhere [5], only a brief overview of just the key functions and components are given in this paper.

The hydride beds contained the alloy powders in welded cylindrical pressure vessels fabricated from 316L stainless steel (SS). Figure 2 gives schematic cross-sectional views of the three beds along with their key performance parameters and requirements. The radial geometries are used to enhance the hydrogen and heat flows in the powder beds for the indicated transfer conditions. Tubular filters of porous 316L SS with 2 micron pore dimensions confine the powder in the allocated regions indicated in Figure 2. The removal of the heat released during hydrogen absorption by the alloys is paramount to the collection of the quantities of gas at the designated pressures and reaction times. Since the heat transfer requirements are especially demanding for the fast absorption bed, the hydride powder is equally distributed among seven tubes which have copper fin assemblies brazed to the tube walls as shown in Figure 3. These tube assemblies are inserted into an aluminum housing as indicated in Figure 2. The spaces between the aluminum housing and the seven copper-finned tubes contain the paraffin wax n-hexadecane to extract up to 157.3 kJ of the heat of reaction from hydrogen absorption using the latent heat of fusion as this previously frozen phase change material (PCM) melts.

The high pressure and low pressure hydride containers are surrounded by 3.8 mm annular volumes that serve as gas-gap heat switches by giving thermal isolation when evacuated and allowing heat conduction to the radiators when filled with helium at nominal 6.7 kPa pressure. Figure 4 illustrates how the three heaters and thermocouples

are arranged on the outer wall of the hydride tubes (i.e., inner surface of the gas gaps) of the low and high pressure beds. The bellows at the ends allow for component expansion and contraction during the temperature cycling. An outer housing consisting of a 316L SS/aluminum/316L SS inertia-welded structural form with mounting interfaces for the radiator was welded to the stainless steel bellows at both ends to form the gas gap. The photograph in Figure S shows the three completed hydride beds (without the radiators) attached to the baseplate in approximately the same orientation as given in Figure 1.

Although the individual steps in the assembly process varied considerably from bed to bed due to their different configurations, many similar procedures were used to ensure cleanliness and structural integrity. All handling and processing of the sorbent alloy powders (including filling the beds) were performed in an argon atmosphere glove box system that maintained oxygen and moisture levels below 1.0 and 0.1 ppm, respectively. The porous filter tubes were subjected to vacuum bakeouts and argon purges at temperatures up to 625 K. Electron beam welding was used to seal the sorbent beds after the powders were added. The welding chamber was flushed and back filled with argon and during evacuation it was isolated from the diffusion pump by a liquid nitrogen cold trap. Helium leak tests verified that no detectable leaks were present after the closure welds and after the high pressure proof tests were performed. Prior to initial exposures of the alloys to hydrogen gas, each bed was given a thorough bakeout using an oil-free turbomolecular vacuum pump station while monitoring the evolved gases (e.g., H₂O, CO₂, CH₄, hydrocarbons, and pump oils) with a residual gas analyzer. Although the low and high pressure beds were heated up to 585 K during these vacuum bakeouts, the fast absorption bed was only heated to 365 K since it also contained the liquid n-hexadecane PCM that could either degrade or generate excessive pressures if heated above about 400 K. After bakeout, each bed at room temperature would pump to below 10⁻⁶ torr. Furthermore, the alloy in each bed immediately reacted with hydrogen upon initial exposure and the hydrogen stoichiometries obtained upon completion of the first reaction closely correlated with previously determined equilibrium contents of well activated material.

SBA TEST RESULTS AND DEXXJSS10N

In order to establish that individual hydride sorbent beds could meet the BETSCE system performance requirements [5] (which are briefly summarized in Figure 2) for the quantities and kinetics of both hydrogen absorption and desorption, a test facility containing appropriately sized calibrated reference volumes and pressure sensors was assembled at Aerojet. A computer operated control system was used to provide the desired amounts of hydrogen that would simulate a broad range of the operating conditions expected for each sorbent bed in the BETSCE periodic cycle [3-5]. Because the flight radiators are ineffective under ambient laboratory conditions, aluminum plates with brazed aluminum tubes were mounted at the radiator interfaces to the sorbent beds. A temperature regulated refrigerated bath/recirculator pumped a water/ethylene glycol solution through the tubes to provide the desired cooling. A separate gas manifold was built to provide either helium gas or vacuum using an oil-free pump to replicate the conditions for the flight operation of the helium gas gap switch. Conventional laboratory electronic equipment was used to power the bed heaters and PRTs and record the current and voltage readings during testing. All data were collected and analyzed with Labview software on a Macintosh Quadra computer.

The fast absorption bed (FAB) had been filled with 922.6 grams of LaNi_{4.8}Sn_{0.2} powder in the seven Cu-finned tubes and with 663.5 grams of n-hexadecane as the phase change material. After heating the FAB to 363 K for several hours during a thorough

vacuum bakeout and performing two activation cycles (i.e., hydrogen absorption followed by vacuum desorption), the bed was cooled below 280 K to freeze the PCM. Upon subsequent exposure to hydrogen, the cooled FAB absorbed a total of 12.5 grams of hydrogen at a final pressure of 4.0 atm, with 11.0 grams absorbed in the first 40 seconds. The BETSCE performance goal specified [4,5] for the FAB is to absorb 8.0 grams of hydrogen within 80 s while maintaining the pressure below 2.5 atm. Figure 6 presents the hydrogen pressure and the FAB outer surface temperatures at several locations that were obtained during an absorption test in which 9.4 grams of hydrogen is supplied at a constant flow of 0.117 g/s between the times denoted by A and B. As the hydrogen was being absorbed, the FAB wall temperature rose above the 289 K melting point for n-hexadecane. As shown in Figure 6, the FAB was able to maintain the pressure below 2.5 atm. When the hydrogen flow was stopped at time B, the pressure rapidly fell. Similar behavior was observed during all the other absorption tests in which 6.0 to 9.4 grams of hydrogen flowed into the FAB during 80s intervals. The test results demonstrate that the FAB is capable of rapidly absorbing the hydrogen from the BETSCE J-T cryostat during the brief cool down period and as the liquid phase accumulates at a temperature below 25 K in the J-T cold tip reservoir. When the FAB was heated to 350-365 K for periods of 30-75 minutes, sufficient quantities of hydrogen were desorbed at pressures between 2.5-3.0 atm that the bed returned to its starting composition for another absorption test.

The low pressure bed (LPB), which contains 228.5 grams of ZrNi powder, absorbed 4.57 grams of hydrogen [immediately] after its activation treatment. However in accordance with the equilibrium pressure-composition isotherms obtained by Luo, et al. [6] for $ZrNiH_x$ with $x < 1.0$ approximately 1.75 grams of hydrogen cannot be removed from the LPB during the BETSCE procedure of heating the bed to 575-590 K for its desorption at pressures in the range of 0.5- 1.0 atm. Furthermore, the increase in the $ZrNiH_x$ equilibrium pressure [6] when $x > 2.3$ limits the amount of hydrogen that the LPB can absorb before exceeding the BETSCE goal of maintaining the pressures over solid hydrogen to below 2.0 atm. Hence, the maximum reversible capacity of the LPB during conditions corresponding to operation in a BETSCE cycle [4,5] is about 2.0 grams of hydrogen which reflects the composition change in $ZrNiH_x$ over the range $1.0 < x < 2.3$.

Results from a representative two-step absorption test of the activated LPB are summarized in Figure 7. The pressure behavior is shown by portions obtained using two gauges with very different ranges due to the nearly four orders of magnitude change during this experiment. In the initial step, 0.55 grams of hydrogen at a starting pressure of 2.0 atm is admitted to the LPB, which in the 40s between points A and B in Figure 7 absorbs nearly all this gas to give a residual pressure of 0.5 torr (i.e., 0.067 kPa) at point R. A constant hydrogen flow at 5.44×10^{-4} g/s is then started at time B and continues for 20 minutes. Figure 7 shows how the LPB maintains the pressure at a nearly constant 1.5 torr (0.20 kPa) as this additional 0.65 grams of hydrogen is being absorbed. The bed temperatures are shown to rise an additional 3-5 K above the increase produced during the initial 40s burst absorption stage. Similar behavior was observed during other two-step absorption tests with up to 0.74 grams of hydrogen being absorbed by the LPB during the first 40s burst step. The BETSCE performance goals for the LPB are absorption of up to 0.7 grams of hydrogen within 40s with the pressure falling to below 0.2 kPa followed by absorption of an additional 0.6 grams during the next 10 minutes at a pressure not above 0.2 kPa. The results shown in Figure 7 clearly demonstrate that the LPB can quickly absorb hydrogen at sufficiently low pressure to satisfy these goals and will form solid hydrogen at 10 K when integrated into the BETSCE cryocooler. When heated up to 588 K, the LPB desorbed between 1.0 and 1.9 grams of hydrogen

(depending upon the amounts absorbed by the bed during preceding absorption tests) at pressures around 1.0 atm.

The high pressure bed (HPB) has been filled with 1.488 Kg of $\text{LaNi}_{4.8}\text{Sn}_{0.2}$ powder as the sorbent alloy and gave an initial total hydrogen storage capacity of 19.4 grams upon activation. In contrast to the very demanding requirements imposed during hydrogen absorption by the FAB and LPB to permit formation of solid hydrogen within 2 minutes, the conditions for the two absorptions by the HPB are rather modest. The laboratory simulation tests showed the HPB to be quite capable of meeting the BETSCE goals of first absorbing less than 1.5 grams in 30 minutes at pressures between 0.5- 1.0 atm, followed by absorbing at least 8.0 grams in 60 minutes at a nominal 1.5 atm pressure. Desorption tests with the HPB successfully settled a critical issue whether the HPB could transfer a sufficient quantity (i.e., > 9.0 grams) of hydrogen at a nominal 100 atm pressure into the BETSCE storage volume when the heaters had limited power (i.e., about 180 watts) available. The response of the HPB when heated at 179 watts is summarized in Figure 8. The temperatures are for three locations on the inner wall and reveal considerable gradients. Between times A and B the HPB is only open to a small volume line with a high pressure transducer to record the de-sorption during heating. At time B, the HPB is opened to a 4.5 liter volume previously filled with hydrogen at 70 atm, which is also the pressure generated within the heated HPB and its line at this time. Thus, the pressure increase from B to C in Figure 8 corresponds to hydrogen transfer into the standard volume until the pressure reaches 102.5 atm. This experiment, in which 11.6 grams of hydrogen were desorbed in less than 3 hours, simulates the refilling of the BETSCE volume at the end of the cool down cycle [4]. Other desorption experiments generated nominal 100 atm pressure with the transfer of 10.9 -12.2 gmms of hydrogen over 3-4 hours. Hence, the HPB produced final pressures of 100 atm and exceeded the BETSCE performance goal of transferring at least 9.35 grams into the storage volume within allotted times for nominal 180 watts of heating, power.

CONCLUSIONS

The three metal hydride sorbent beds for the BETSCE project have been fabricated and tested at Aerojet under various flight simulation conditions. The reversible hydrogen storage capacity and absorption-desorption kinetics for each individual bed exceeded the design goals that had been specified [4,5] to allow for the production of solid hydrogen within 2.0 minutes from the initiation of a BETSCE rapid cool down sequence. The completed BETSCE sorbent bed assembly was delivered to JPL in October, 1993. The SBA has been integrated with the other BETSCE components for system level functional testing at JPL. Initial laboratory tests [7] showed that hydrogen absorption by the FAB led to the formation of liquid within 40s after the start of the cool down from a 70 K J-T coldstage temperature. Solid hydrogen at $T < 11$ K was subsequently achieved within 95 s from the start as the LPB absorbed the hydrogen gas evolving from the J-T cryostat. A minimum temperature of 9.56 K has been obtained and the cold stage remained below 11 K for 20 minutes. The SBA also transferred the hydrogen gas back into the storage reservoir for repeated blowdown experiments. These results confirmed that the three metal hydride sorbent beds were meeting (or exceeding) all of the original BETSCE performance objectives.

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FIGURE CAPTIONS

Figure 1. Overview of the BETSCE Sorbent Bed Assembly (SBA) on its mounting baseplate.

Figure 2. End views of the SBA metal hydride beds with brief summaries of their 10 K BETSCE performance requirements.

Figure 3. Components for one of the hydride containing tubes with copper fins and porous filter that comprise the fast absorption bed.

Figure 4. The hydride containing portion of the high pressure bed with heaters and thermocouples attached. The low pressure bed appeared nearly the same at this stage of assembly.

Figure 5. The three metal hydride beds are shown mounted on the baseplate without the radiators.

Figure 6. The behavior of the fast absorption bed during a representative absorption run as 9.4 grams of hydrogen was absorbed in 80 seconds.

Figure 7. The results obtained from the low pressure bed during a representative two step hydrogen absorption experiment.

Figure 8. The behavior of the high pressure bed during, a representative desorption run,

FIGURE 1

FIGURE

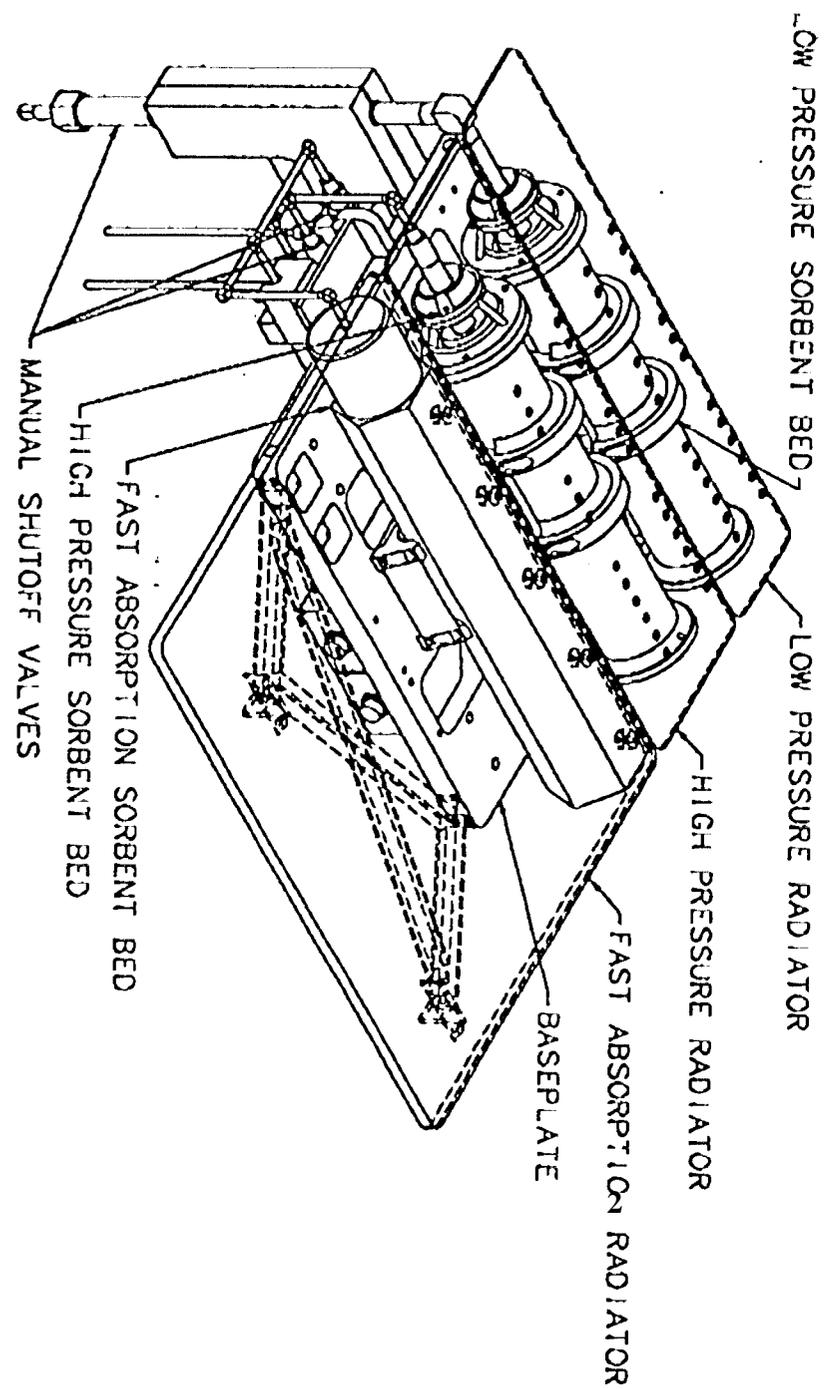


FIGURE 2

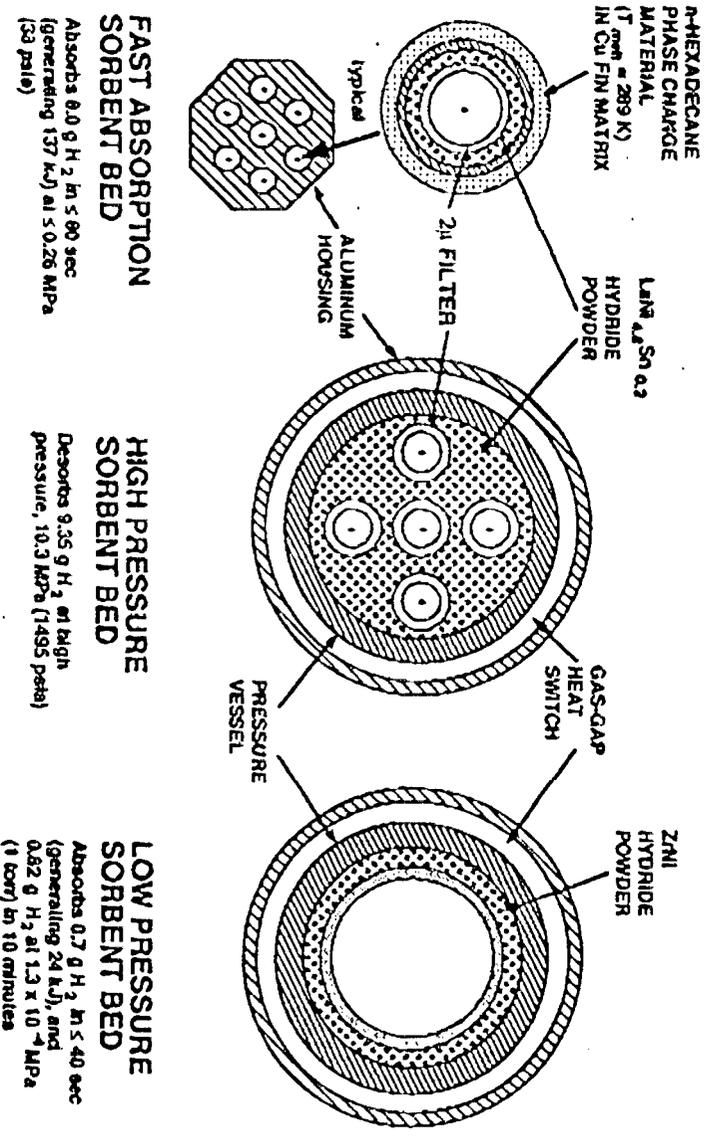
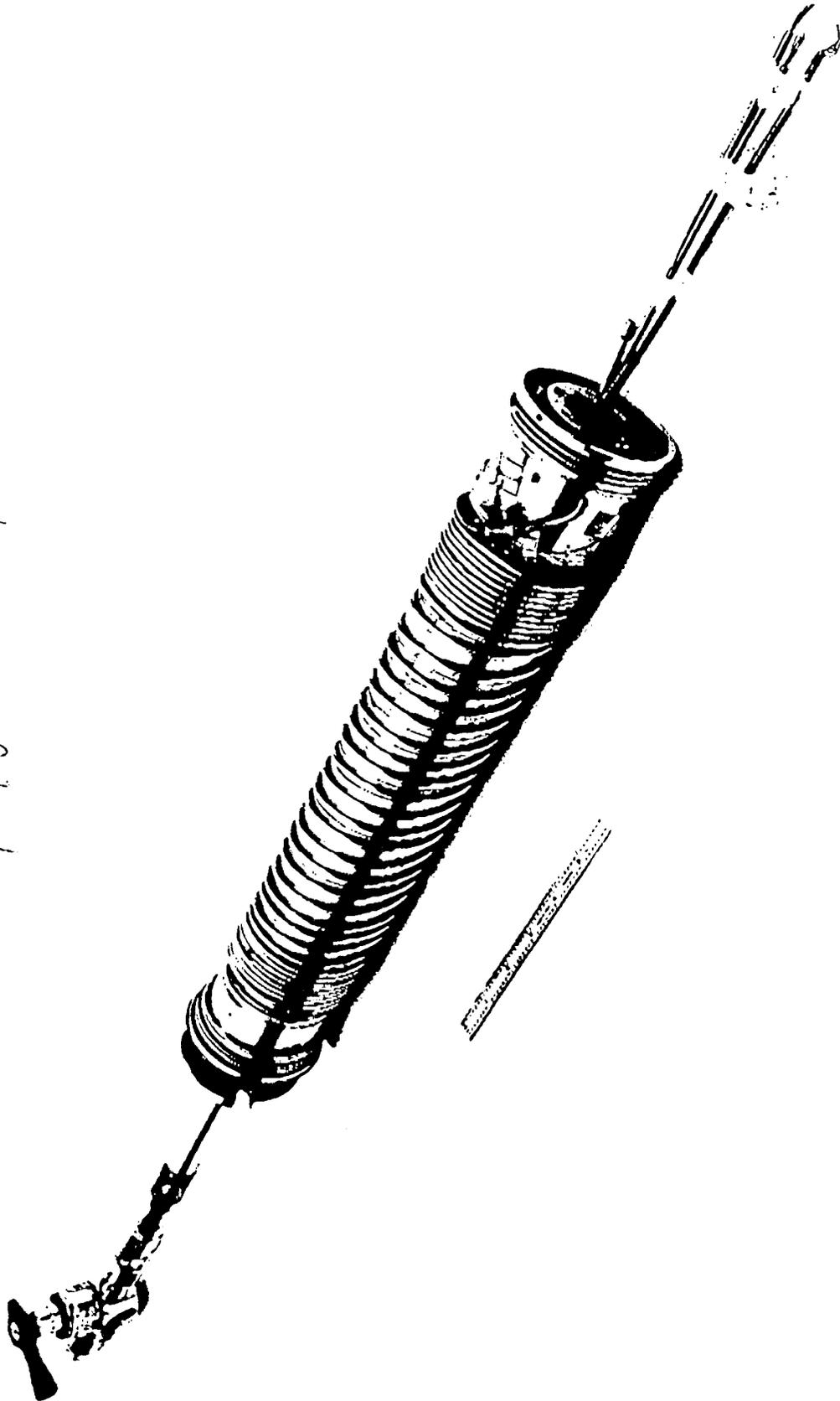


FIGURE 3



FIGURE 4



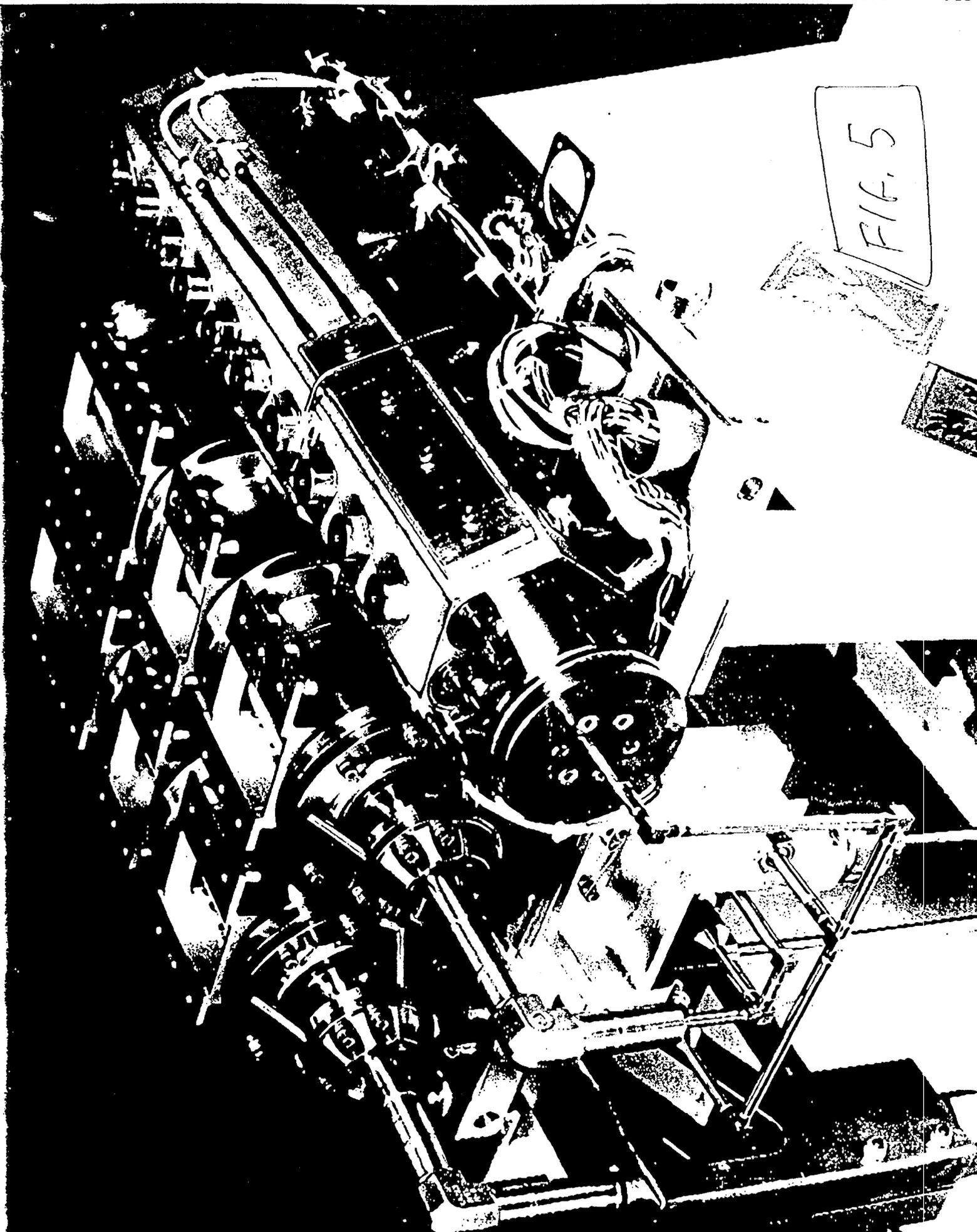


FIG. 5

FIGURE 6

FAB Fast Absorption 80 sec. 9/14/93 Run #2 Plotted 3/10/94

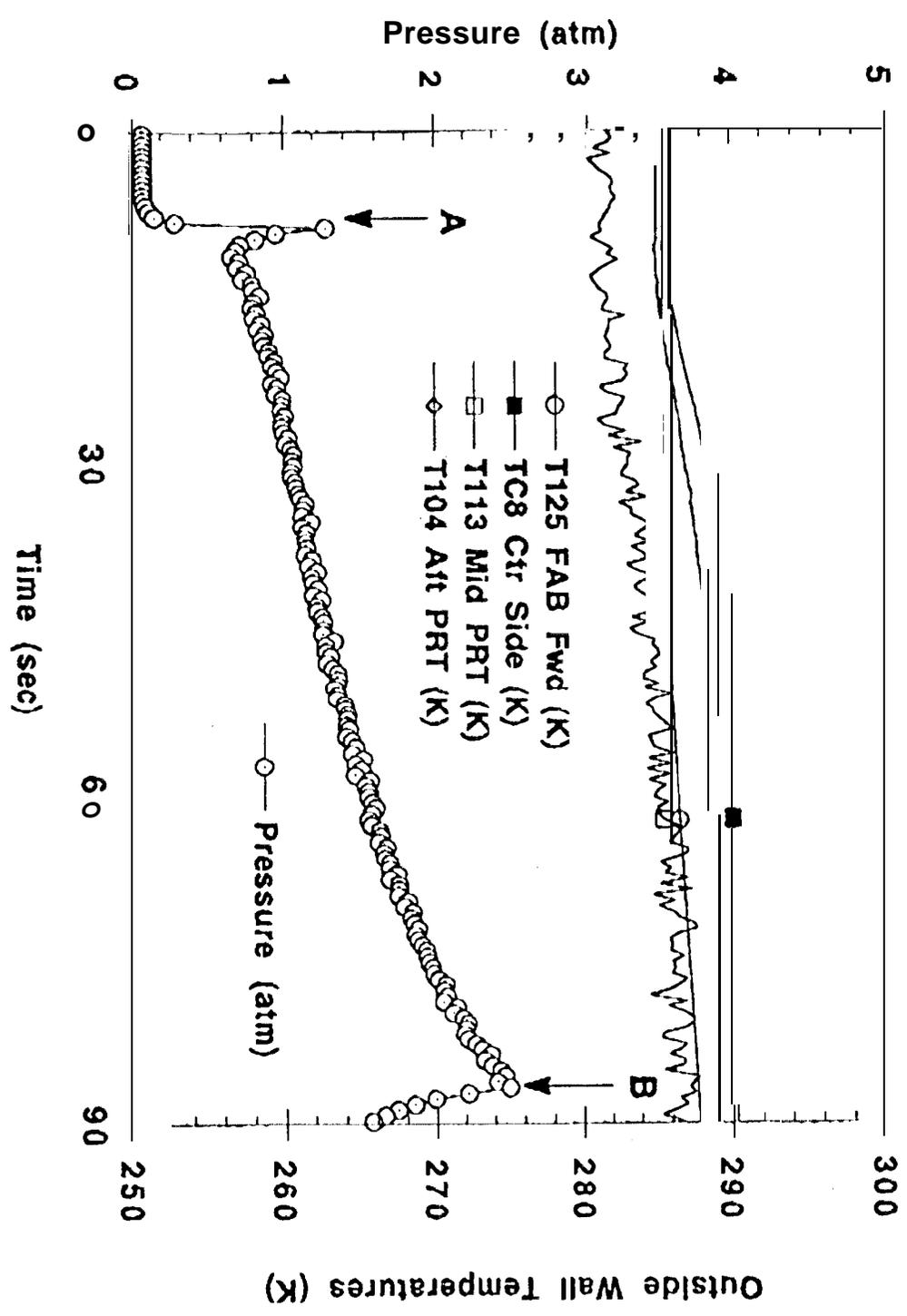


FIGURE 7

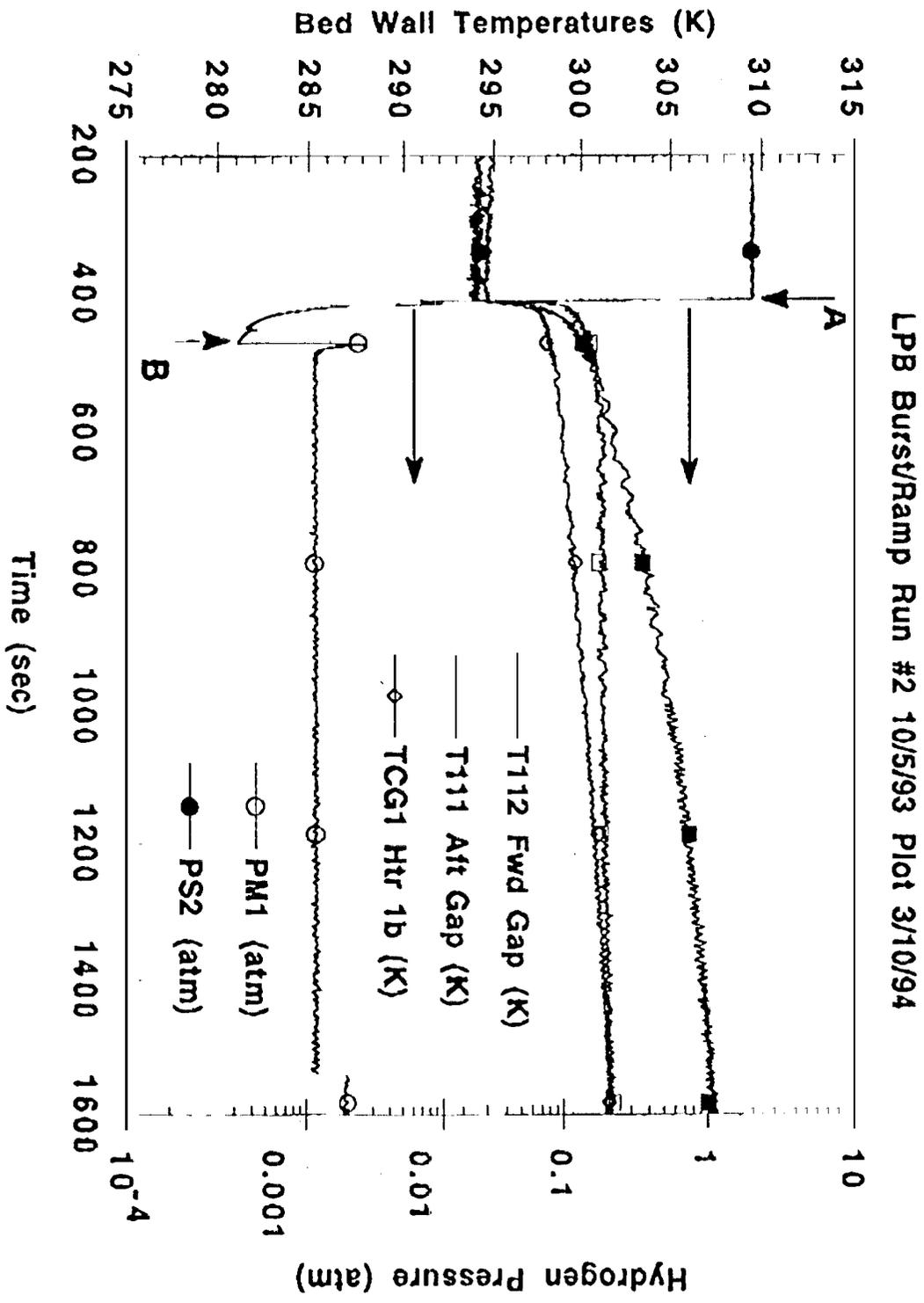


FIGURE 8

