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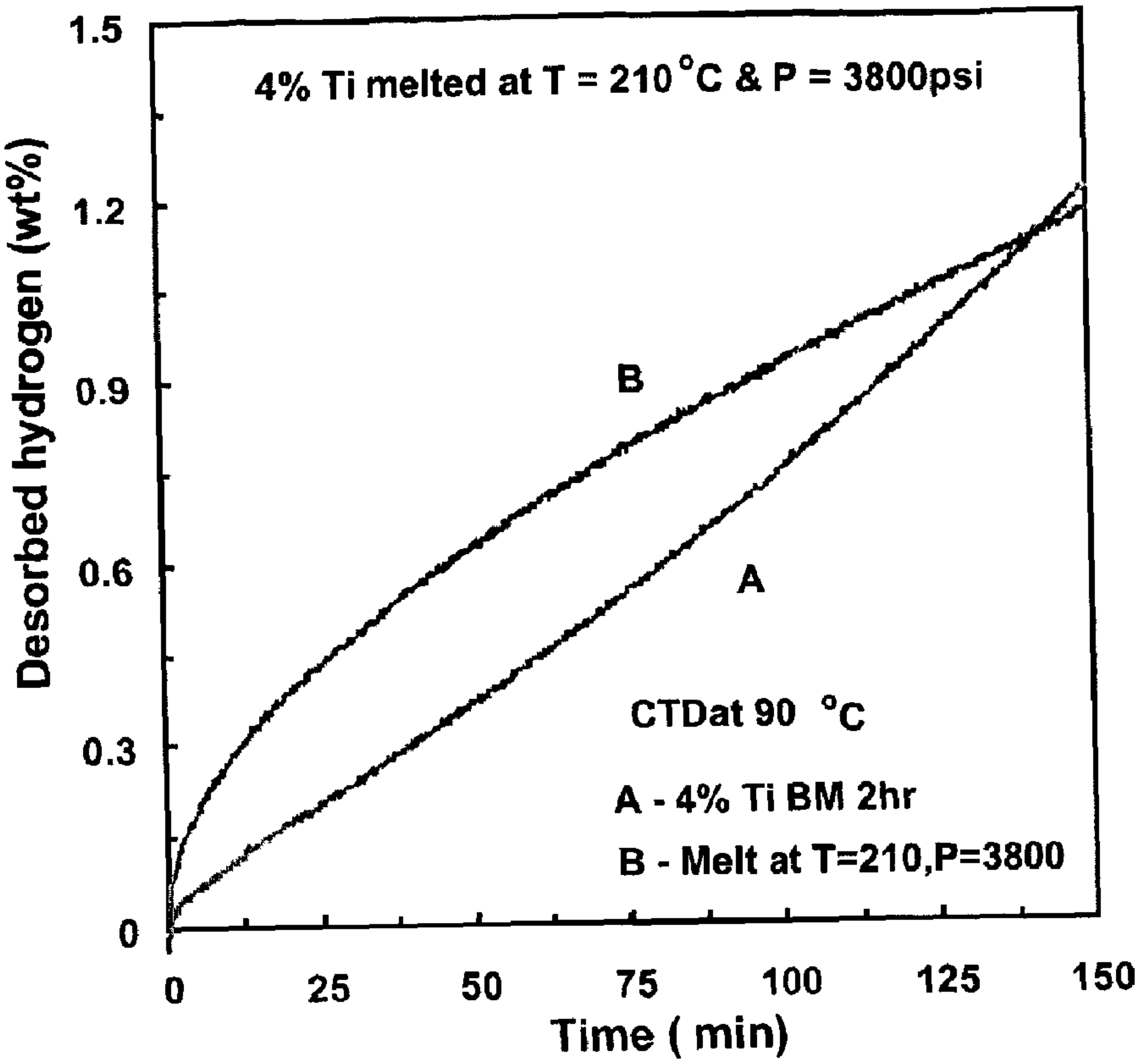
(54) Title: COMPLEX HYDRIDES FOR HYDROGEN STORAGE



(57) Abrégé/Abstract:
A hydrogen storage material and process of forming the material is provided in which complex hydrides are combined under conditions of elevated temperatures and/or elevated temperature and pressure with a titanium metal such as titanium butoxide. The resulting fused product exhibits hydrogen desorption kinetics having a first hydrogen release point which occurs at normal atmospheres and at a temperature between 50°C and 90°C.

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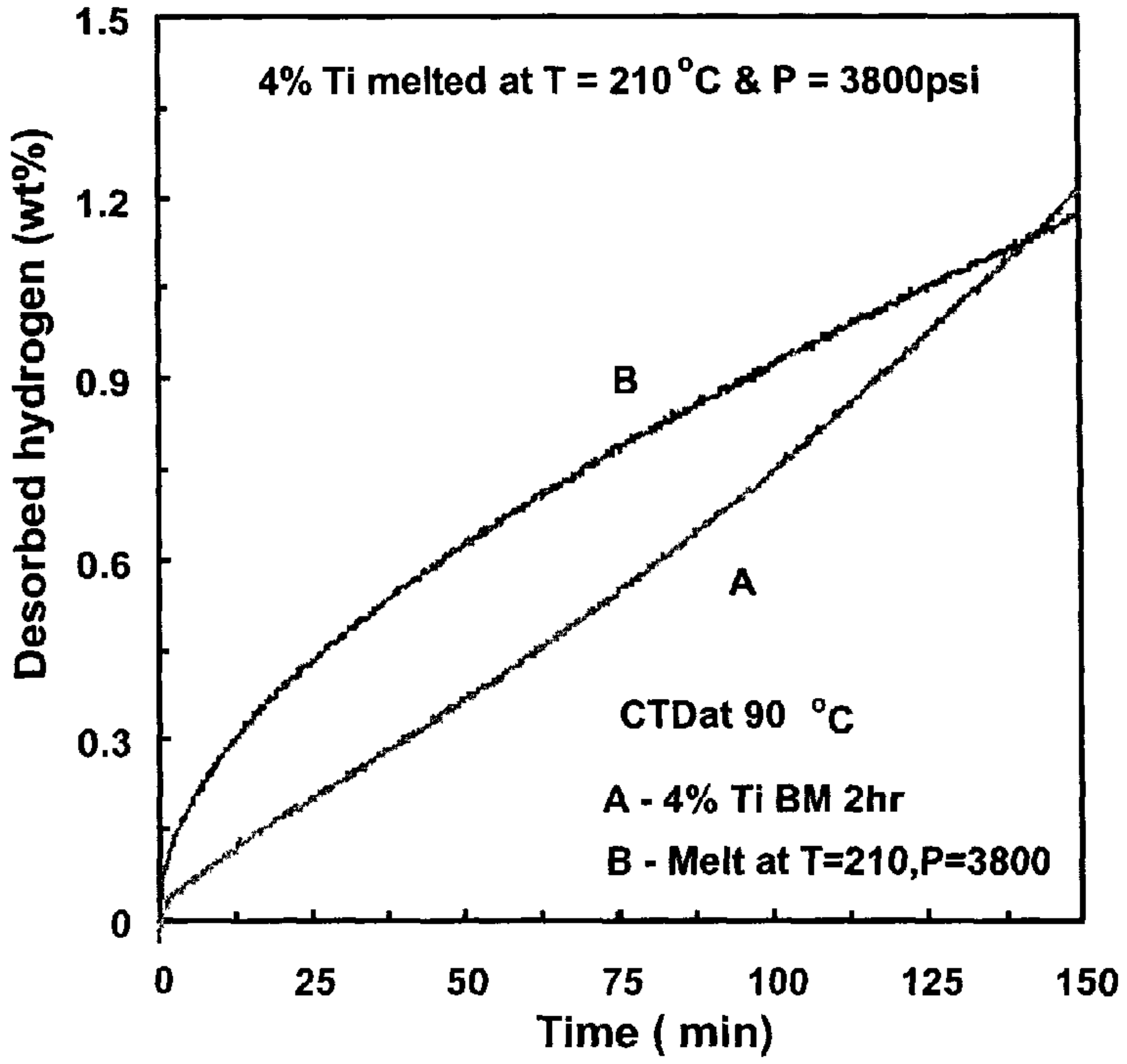
(54) Title: COMPLEX HYDRIDES FOR HYDROGEN STORAGE



(57) Abstract: A hydrogen storage material and process of forming the material is provided in which complex hydrides are combined under conditions of elevated temperatures and/or elevated temperature and pressure with a titanium metal such as titanium butoxide. The resulting fused product exhibits hydrogen desorption kinetics having a first hydrogen release point which occurs at normal atmospheres and at a temperature between 50°C and 90°C.

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COMPLEX HYDRIDES FOR HYDROGEN STORAGE

STATEMENT AS TO RIGHTS TO INVENTIONS MADE UNDER FEDERALLY SPONSORED RESEARCH AND DEVELOPMENT

This invention was made with Government support awarded by the United States Department of Energy. The Government has certain rights in the invention.

FIELD OF INVENTION

The present invention relates generally to the field of a reversible hydrogen storage material. More particularly, the present invention relates to metal hydrides. In particular, the invention relates to complex metal hydride materials which have been melted or heated near the material's melting point, under a combination of temperature and pressure, so as to provide a unique hydrogen storage material and a method for making the same.

BACKGROUND OF THE INVENTION

Hydrogen has long been proposed as an optimal fuel for transportation needs due to its abundance as well as its environmentally friendly properties. To date, the use of hydrogen as a fuel source has been limited by difficulties in providing adequate hydrogen storage capabilities, particularly for vehicular use. Heretodate, the primary methods of hydrogen storage involve storage as a compressed gas in pressurized tanks or utilizing low temperature storage as liquid hydrogen. Such storage mechanisms are impediments to vehicular use of hydrogen fuel, since high pressure and cryogenic storage technology are impractical for vehicular use. As a result, there have been extensive efforts to develop hydrogen storage systems using materials which offer the combination of high density hydrogen storage capabilities, favorable hydrogen disssociation kinetics, and using materials and processes having sufficiently low costs to be feasible for commercial transportation applications.

For instance, it is known in the art that the kinetics of hydrogen desorption from some alanates can be enhanced by doping an alanate such as sodium aluminum hydride with a transition metal. Sodium aluminum hydride has poor hydrogen storage kinetics and is reversible only under severe conditions of temperature and/or pressure change. Recently, it has been established that titanium doping of $NaAlH_4$ can enhance the kinetics of hydrogen desorption and can provide for more moderate conditions for dehydriding. Work by Bodanovic and Schwickardi, as described in U.S. Pat. No. 6,106,801, provides for titanium wet doping of $NaAlH_4$ using an ether suspension have a 2 mole percent of titanium tetra-n-butoxide $(Ti(OBu)_4$ However, the temperatures and kinetics of hydrogen adsorption and desorption of the doped material are such that the material still remains impractical for transportation applications.

U.S. Pat. No. 6,074,453 (assigned to Iowa State University Research Foundation, Inc.), discloses a method for making a hydrogen storage powder which is gas atomized under high temperatures and pressures to form generally spherical powder particles. The powder exhibits a small particle size which is stated to be resistant to microcracking during hydrogen adsorption/desorption cycling. However, the '453 reference utilizes hydrogen storage materials such as $LaNi_5$ and other similar AB_5 type materials which are too expensive for widespread use in transportation needs. Additionally, the resulting hydrogen storage powder set forth in the '453 patent requires substantial temperature and pressure variations in order to bring about useful adsorption and desorption cycles.

There remains a need for hydrogen storage materials that have a useful hydrogen storage capacity combined with low stringency release kinetics. Accordingly, there remains room for variation and improvement within the art of hydrogen storage materials.

SUMMARY OF THE INVENTION

It is one aspect of one of the present embodiments to provide a hydrogen storage material which can achieve a release of stored hydrogen at a temperature of about 80-90°C and demonstrate practical kinetics.

It is yet another aspect of at least one of the present embodiments to provide a mixture of a sodium aluminum hydride ($NaAlH_4$) with a small percentage such as about 0.5 to about 5.0 percent by weight of titanium or other transition metals. The sodium aluminum hydride and titanium form, under conditions of heat and pressure, a substantially homogeneous material having the ability to absorb about 5.2 percent by weight of hydrogen, a portion of the stored hydrogen being releasable at a temperature of about 50°C to about 90°C.

It is yet another aspect of at least one of the present embodiments to provide a hydrogen storage material which results from the temperature and pressure fusion of transition metals with a complex hydride, thereby forming a homogeneous melted material having lower temperature hydrogen desorption kinetics.

It is yet another aspect of at least one of the present embodiments to provide a hydrogen storage material which results from the combining of a transition metal near a melting point of the complex hydride which, when cooled, has hydrogen storage properties including low temperature hydrogen desorption kinetics.

It is yet another aspect of at least one of the present embodiments of the invention to provide a hydrogen storage material comprising a high pressure melted fusion of an alanate selected from the group consisting of alkali-metal hydrides, complex metal hydrides, and combinations thereof, and a metal dopant selected from the group consisting of groups III through V transition metals, lanthanum metal complexes, iron, nickel, rare earth metals and combinations thereof. The metal dopants may include alcoholates, alkoxides, halides, hydrides, and organometallic and intermetallic compounds of the referenced metal dopants.

It is yet another aspect of at least one of the present embodiments to provide a hydrogen storage material formed by a pressurized melt, the resulting melt product having useful thermal desorption properties which occur at or about 50°C, indicative of kinetic enhancement of hydrogen sorption properties. Mixtures of different alanates, alanates and borohydrides, and alanates and different elements can be fused near or at the melting point of the mixtures or the melting point of one of the elements in the mixture. The mixture enables substitution of elements among the precursor reagents or results in the formation of a new composition(s).

In yet another aspect, there is provided a process for forming a hydrogen storage material comprising providing a sodium aluminum hydride; mixing a 1%/wt of titanium to said sodium aluminum hydride; and supplying a combination of heat and pressure in the presence of hydrogen gas sufficient to melt said sodium aluminum hydride and titanium mixture, thereby providing a fused hydrogen storage material having a hydrogen release point at normal atmospheric pressure of between about 50°C to about 90°C.

In yet another aspect, there is provided a process of forming a hydrogen storage material comprising supplying at least one complex hydride selected from the group consisting of hydrides having the formula of $M_y(BH_{4+z})_x$ where M is sodium, calcium, magnesium, zirconium, or iron; B is aluminum or boron; X has a value of between 1 and 4; Y has a value of between 1 and 6; and Z has a value of 0 or 2; mixing with said complex hydride a dopant selected from the group consisting of titanium, zirconium, vanadium, iron, cobalt, nickel, lanthanum, and mixtures thereof; subjecting said mixture of complex hydride and said dopant under pressure in the presence of hydrogen gas; raising the temperature of said mixture of said complex hydride and said dopant and said hydrogen gas to a melting point of said complex hydride; and maintaining said temperature and pressure for a time sufficient to form a fused product, wherein said fused product has a reversible ability to store and release hydrogen.

In yet another aspect, there is provided a process of forming a hydrogen storage material comprising supplying at least one complex hydride wherein said at least one complex hydride comprises lithium hydride; mixing with said complex hydride a dopant selected from the group consisting of titanium, zirconium, vanadium, iron, cobalt, nickel, lanthanum, and mixtures thereof; subjecting said mixture of complex hydride and said dopant under pressure in the presence of hydrogen gas; raising the temperature of said mixture of said complex hydride and said dopant and said hydrogen gas to a melting point of said complex hydride; and maintaining said temperature and pressure for a time sufficient to form a fused product, wherein said fused product has a reversible ability to store and release hydrogen.

In yet another aspect, there is provided a process of forming a hydrogen storage material comprising supplying at least one complex hydride wherein said at least one complex hydride comprises sodium hydride; mixing with said complex hydride a dopant selected from the group consisting of titanium, zirconium, vanadium, iron, cobalt, nickel, lanthanum, and mixtures thereof; subjecting said mixture of complex hydride and said dopant under pressure in the presence of hydrogen gas; raising the temperature of said mixture of said complex hydride and said dopant and said hydrogen gas to a melting point of said complex hydride; and maintaining said temperature and pressure for a time sufficient to form a fused product, wherein said fused product has a reversible ability to store and release hydrogen.

In yet another aspect, there is provided a process of forming a hydrogen storage material comprising supplying at least one complex hydride wherein said at least one complex hydride comprises a mixture of sodium aluminum hydride, lithium hydride, and sodium hydride; mixing with said complex hydride a dopant selected from the group consisting of titanium, zirconium, vanadium, iron, cobalt, nickel, lanthanum, and mixtures thereof; subjecting said mixture of complex hydride and said dopant under pressure in the presence of hydrogen gas; raising the temperature of said mixture of said complex hydride and said dopant and said hydrogen gas to a melting point of said complex hydride; and maintaining said temperature and pressure for a time sufficient to form a fused product, wherein said fused product has a reversible ability to store and release hydrogen.

These and other features, aspects, and advantages of the present invention will become better understood with reference to the following description and appended claims.

BRIEF DESCRIPTION OF THE DRAWINGS

A full and enabling disclosure of the present invention, including the best mode thereof, to one of ordinary skill in the art, is set forth more particularly in the remainder of the specification, including reference to the accompanying drawings.

Figure 1 sets forth hydrogen desorption kinetics of a prior art hydrogen storage material, $NaAlH4$ with 1 percent titanium prepared by a conventional ball milling (BM) process using sodium aluminum hydride ($NaAlH4$) and a titanium metal such as titanium butoxide.

Figure 2 is a hydrogen desorption graph of a fused mixture of $NaAlH4$ with a 1 percent titanium butoxide according to the present invention.

Figures 3A and 3B set forth data for thermogravimetric analysis of samples of NaAlH4 doped with 4% TiCl3 prepared by the fusing process of the present invention (3B) and compared to a control sample (3A) prepared only using a ball milling (BM) process.

Figure 3C sets forth data from a constant temperature desorption (CTD) analysis using the materials described in reference to Figures 3A and 3B.

Figure 4A is an x-ray defraction pattern of a sample of equimolar mixtures of NaHLiH, and $NaAlH_4$ dry mixed with a mortar and pestle.

Figure 4B is an x-ray defraction analysis of the material seen in Figure 4a following fusion using heat and pressure.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Reference now will be made in detail to the embodiments of the invention, one or more examples of which are set forth below. Each example is provided by way of explanation of the invention, not limitation of the invention. In fact, it will be apparent to those skilled in the art that various modifications and variations can be made in the present invention without departing from the scope or spirit of the invention. For instance, features illustrated or described as part of one embodiment can be used on another embodiment to yield a still further embodiment. Thus, it is intended that the present invention covers such modifications and variations as come within the scope of the appended claims and their equivalents. Other objects, features, and aspects of the present invention are disclosed in the following detailed description. It is to be understood by one of ordinary skill in the art that the present discussion is a description of exemplary embodiments only and is not intended as limiting the broader aspects of the present invention, which broader aspects are embodied in the exemplary constructions.

In describing the various figures herein, the same reference numbers may be used throughout to describe the same material or process pathway. To avoid redundancy, detailed descriptions of much of the materials or processes once described in relation to a figure or an embodiment may not be repeated in the descriptions of subsequent figures or embodiments, although such material or process may be identified with the same reference numbers.

The present invention provides for a product and a process for producing a product in the form of novel materials formed under melting conditions formed by a combination of heat and pressure. A metal complex hydride in combination with a transition metal, a mixture of other complex hydrides and/or other elements are subject to the melt conditions. The resulting cooled material, hereinafter referred to as a “fused” or “hybrid” product, results in a hydrogen storage material having improved properties with respect to hydrogen storage capacity and hydrogen release kinetics compared to prior art hydrogen storage materials prepared from physical ball milling techniques and/or chemical treatment techniques. Additionally, the fused products exhibit excellent performance of repeated cycles of hydrogen adsorption and desorption.

Alternatively, fused or hybrid products can be formed by bringing the reactants under temperature and pressure a few degrees below a melting point of at least one of the mixture components. Under these conditions, it is believed that various elements within the mixture components may substitute with one another so as to bring about a resulting novel fused or hybrid product having beneficial hydrogen storage capacity and release kinetics.

While not wishing to be limited by theory, it is believed that the resulting fused product achieves an enhanced distribution and uniformity of materials. The resulting fused product exhibits excellent physical stability and has desirable hydrogen adsorption and release kinetics.

As a result of the improved thermodynamics of the fused product, lower temperature changes can be used to bring about a release of stored hydrogen. The observed improvement in kinetics represents a fundamental advance in capabilities of alanate-based hydrogen storage materials. Additionally, the ability to form fused products allows for materials having enhanced amounts of dopants which are believed to offer even further improvements in the hydrogen storage kinetics. It is believed that the fusion of dopant metals such as transition metals with complex hydrides results in a fused product having improved hydrogen storage properties. The improvements are believed attributable to the high mobility of atoms which occurs at or near the melting state of the complex hydride and which results in a more homogeneous product.

Example 1

One example of a fused hydrogen storage product is provided by the reaction product of one gram of sodium aluminum hydride mixed with 1 to 2 milliliters of 100mM titanium butoxide in tetrahydrofuran (THF), the volume of titanium butoxide adjusted to achieve a 1 percent by weight target amount of titanium. The two components were mixed and stirred under an inert atmosphere in a glovebox using an agate mortar and pestle until the sample was dried.

The dried sample is placed in a pressure bomb and sealed prior to removal from the glovebox. Outside the glovebox, the bomb is connected to a hydrogen supply line and a separate vacuum line in a “T” configuration. Hydrogen is cycled through the lines to purge air prior to pressurization of the pressure bomb. Following purging, the pressure bomb is opened for pressurization with hydrogen gas to a pressure of about 3700 psi (260 atmosphere). The pressure bomb is then sealed at the elevated pressure and disconnected from the hydrogen supply line.

The pressure vessel is placed inside a heating mantle and brought to a temperature of about 190°C to about 220°C for an interval of at least 3 hours. The addition of heat achieves an internal bomb pressure of up to about 5000 psi (353 atmosphere). Following heating, the pressure vessel is cooled to room temperature and then depressurized. Depressurization occurs in an inert atmosphere glove box where the resulting melted material is removed. 20 mg samples are removed for analysis using thermogravimetric techniques, the results of which are set forth in Figure 2.

As seen in Figure 2, the fused hydrogen storage material exhibit three distinctive hydrogen release points. The first point occurs at a point beginning at about 50°C and achieving a more preferable release at between about 80-90°C. As additionally seen in Figure 2, a second hydrogen release occurs at a temperature between about 140°C to about 150°C with a third release at a temperature of about 190°C to about 200°C. As seen in reference to Figure 2, the weight loss percent of hydrogen at the lowest temperature release peak is approximately 3.2 percent weight loss of hydrogen

Example 2

One gram samples of $NaAlH_4$ doped with 4% $TiCl_3$ were subjected to high intensity ball milling for 2 hours. Following ball milling, the metal hydride was fused by exposure to a temperature of 210°C and a pressure of 3800 psi using hydrogen gas for about 4 hours. At the end of 4 hours, the temperature and pressures were allowed to gradually equilibrate to standard conditions.

Thermogravimetric analysis (TGA) was performed on control samples (ball milled only) and fused samples which were additionally subjected to the combination of hydrogen pressure and elevated temperature. As seen in reference to Figure 3A, fused (melt) demonstrate improved low temperature kinetics of the fused samples as opposed to samples obtained by ball milling. The second curve in Figure 3A is a second temperature program desorption run repeated 24 hours later for the fused product. The kinetics and capacity of the 24 hour delayed run reflects hydrogen desorption from hydrogen which was absorbed overnight.

It should be noted that the evaluated of the fused fused product as represented in Figures 3A and 3B were made under conditions designed to show differences of low temperature kinetics. The total hydrogen capacity of the fused material can be improved by increasing volumes and by adjusting various ratios of metal hydrides, dopants, and catalyst materials so as to enhance hydrogen adsorption/desorption capacity.

Figure 4B is a replicate of the materials and processes described above and conducted on a different day. As seen in Figure 4A, similar kinetics are present for the fused product showing increased desorption kinetics from about 75° to 100°C, and more preferably about 90°C.

Example 3

One gram mixtures of a 1:1:1 mole mixture of NaH LiH and $NaAlH_4$ were prepared by a melt preparation process at a temperature of about 210°C and with a hydrogen pressure of 3800 psi for 4 hours. Prior to the melting process, samples were dry mixed using a mortar and pestle. A sample of the mixture processed only with the mortar and pestle was used as a control sample.

Following the melt preparation process, x-ray defraction patterns were obtained of control samples and the fused/melted samples. Set forth in Figure 4A is the x-ray defraction pattern of control sample as mixed with only a mortar and pestle. As seen in Figure 4A, the beginning constituents are identified along with the stainless steel holder “S” and a transparent tape covering “T” placed over the holder.

Figure 4B sets forth the x-ray defraction patterns of the fused product. As indicated in Figure 4B, appreciable concentrations of Na_2LiAlH_6 and Na_3AlH_6 were formed. The data also indicates that portions of the NaH LiH, and $NaAlH_4$ were left unreacted. It is believed the reaction products identified are from two competing overall reactions including:

$$2NaH + NaAlH_4 = Na_3AlH_6 \qquad (1)$$

$$LiH + NaH + NaAlH_4 = Na_2LiAlH_6 \qquad (2)$$

Observations of repeated hydrogen adsorption and release indicates the melted product exhibits excellent properties in terms of cycling efficiency. These observations suggest the melted reaction product is resistant to loss of structural integrity during repeated cycles of hydrogen pressurization and release.

The ability to create novel fused hydrogen adsorption materials enables one to increase the amounts of titanium and other materials used to form the melted reaction product. The enhanced loading capabilities far exceed prior art loading levels achieved using conventional ball milling or chemical treatment processes. As a result, it is believed that the present process enables categories of wholly new materials which have enhanced storage capabilities and offer improved thermodynamic properties.

It is believed that the present process is useful for a variety of hydrogen storage materials including at least all complex hydrides having the formula of $M_y(AlH_{4+z})_x$ where M is an alkaline, alkaline earth metal or transition metal such as sodium, calcium, magnesium, zirconium, or iron; X has a value of between 1 and 4; Y has a value of between 1 and 6; and Z has a value of 0 or 2. Other complex hydrides useful with the present invention are seen in reference to the general formula of $M_y(BH_{4+z})_x$ where M is the transition metals identified above, B is boron, and X, Y, and Z have the identified values.

To the extent complex hydrides are used with various catalysts or dopants to bring about improved hydrogen storage properties, it is believed that any of the complex hydrides and typically used catalysts can be melted to form a fused reaction product as described above in which the reaction product will offer improved hydrogen storage capabilities and release kinetics. It is believed that pressurized melting or achieving near melting conditions allow for a more effective distribution of materials than is otherwise possible. As a result, conventional proportions of complex hydrides and catalysts may be used to bring about improved properties upon melting the materials. Additionally, it is believed that enhanced levels of catalysts or dopants, as identified in the references as set forth above, may be used including combining different types of dopants such as titanium, zirconium, vanadium, iron, cobalt, nickel, lanthanum, and mixtures thereof. Heretofore, certain of the catalyst metals incorporated into a metal hydride needed to be present in specialized solvents. The present process provides a way of combining the catalyst-like dopants with the complex hydrides which minimizes the need for solvents and allows for enhanced loading levels of the dopants.

In addition to the complex hydrides set forth above, it is also believed that various borohydride complexes such as $NaBH_4$ may also be used in combination with various catalysts and dopants in which the melted product provides for a hydrogen storage material having improved kinetics and hydrogen storage/release properties. In addition, it is noted that the melted materials studied herein were formed by the gradual cooling of the melt. It is envisioned that the cooling process can be changed to include a rapid quenching which may result in a highly non-crystalline structure which may have different hydrogen storage and release properties.

The formation of the fused hydrogen storage material set forth herein is believed particularly useful for forming rapid combinations of various salts so as to form fused complex hydrides. In addition, it is believed beneficial to combine a complex hydride salt or complex hydride forming salts with an organometallics so as to provide a hydrogen absorbing organosalt. Nonlimiting examples of organometallic compounds include titanium IV *tert*-butoxide, and bicyclo compounds.

Additionally, the use of melting point conditions allows one the ability to shape the resulting hydrogen storage fused product into various forms. By way of example, an aluminum bed which heretofore may have been filled with particulates of various hydrogen storage materials can now be filled with a shaped, melted profile conforming to the aluminum bed. This allows for a more efficient packing of the bed and hence increased loading abilities for hydrogen storage with respect to the bed size.

Further, the hydrogen storage fused product described herein is believed to have enhanced catalyst distribution in comparison with materials prepared by traditional ball milling or chemical precipitation. As a result, the enhanced catalyst or dopant loading levels allow for unique reaction products to be formed having improved characteristics with respect to overall hydrogen storage capabilities as well as hydrogen release and adsorption kinetics.

Although preferred embodiments of the invention have been described using specific terms, devices, and methods, such description is for illustrative purposes only. The words used are words of description rather than of limitation. It is to be understood that changes and variations may be made by those of ordinary skill in the art without departing from the spirit or the scope of the present invention, which is set forth in the following claims. In addition, it should be understood that aspects of the various embodiments may be interchanged, both in whole or in part. Therefore, the spirit and scope of the appended claims should not be limited to the description of the preferred versions contained therein.

1. A process for forming a hydrogen storage material comprising:

providing a sodium aluminum hydride;

mixing a 1%/wt of titanium to said sodium aluminum hydride; and,

supplying a combination of heat and pressure in the presence of hydrogen gas sufficient to melt said sodium aluminum hydride and titanium mixture, thereby providing a fused hydrogen storage

material having a hydrogen release point at normal atmospheric pressure of between about 50°C to about 90°C.

2. A process of forming a hydrogen storage material comprising:

supplying at least one complex hydride selected from the group consisting of hydrides having the formula of $M_y(BH_{4+z})_x$ where M is sodium, calcium, magnesium, zirconium, or iron; B is aluminum or boron; X has a value of between 1 and 4; Y has a value of between 1 and 6; and Z has a value of 0 or 2;

mixing with said complex hydride a dopant selected from the group consisting of titanium, zirconium, vanadium, iron, cobalt, nickel, lanthanum, and mixtures thereof;

subjecting said mixture of complex hydride and said dopant under pressure in the presence of hydrogen gas;

raising the temperature of said mixture of said complex hydride and said dopant and said hydrogen gas to a melting point of said complex hydride; and,

maintaining said temperature and pressure for a time sufficient to form a fused product, wherein said fused product has a reversible ability to store and release hydrogen.

3. The process according to claim 2 wherein said at least one complex hydride is sodium aluminum hydride.

4. A process of forming a hydrogen storage material comprising:

supplying at least one complex hydride wherein said at least one complex hydride comprises lithium hydride;

mixing with said complex hydride a dopant selected from the group consisting of titanium, zirconium, vanadium, iron, cobalt, nickel, lanthanum, and mixtures thereof;

subjecting said mixture of complex hydride and said dopant under pressure in the presence of hydrogen gas;

raising the temperature of said mixture of said complex hydride and said dopant and said hydrogen gas to a melting point of said complex hydride; and,

maintaining said temperature and pressure for a time sufficient to form a fused product, wherein said fused product has a reversible ability to store and release hydrogen.

5. A process of forming a hydrogen storage material comprising:

supplying at least one complex hydride wherein said at least one complex hydride comprises sodium hydride;

mixing with said complex hydride a dopant selected from the group consisting of titanium, zirconium, vanadium, iron, cobalt, nickel, lanthanum, and mixtures thereof;

subjecting said mixture of complex hydride and said dopant under pressure in the presence of hydrogen gas;

raising the temperature of said mixture of said complex hydride and said dopant and said hydrogen gas to a melting point of said complex hydride; and,

maintaining said temperature and pressure for a time sufficient to form a fused product, wherein said fused product has a reversible ability to store and release hydrogen.

6. A process of forming a hydrogen storage material comprising:

supplying at least one complex hydride wherein said at least one complex hydride comprises a mixture of sodium aluminum hydride, lithium hydride, and sodium hydride;

mixing with said complex hydride a dopant selected from the group consisting of titanium, zirconium, vanadium, iron, cobalt, nickel, lanthanum, and mixtures thereof;

subjecting said mixture of complex hydride and said dopant under pressure in the presence of hydrogen gas;

raising the temperature of said mixture of said complex hydride and said dopant and said hydrogen gas to a melting point of said complex hydride; and,

maintaining said temperature and pressure for a time sufficient to form a fused product, wherein said fused product has a reversible ability to store and release hydrogen.

7. The process according to claim 6 wherein said sodium aluminum hydride, said lithium hydride, and said sodium hydride are present in equi-molar amounts.

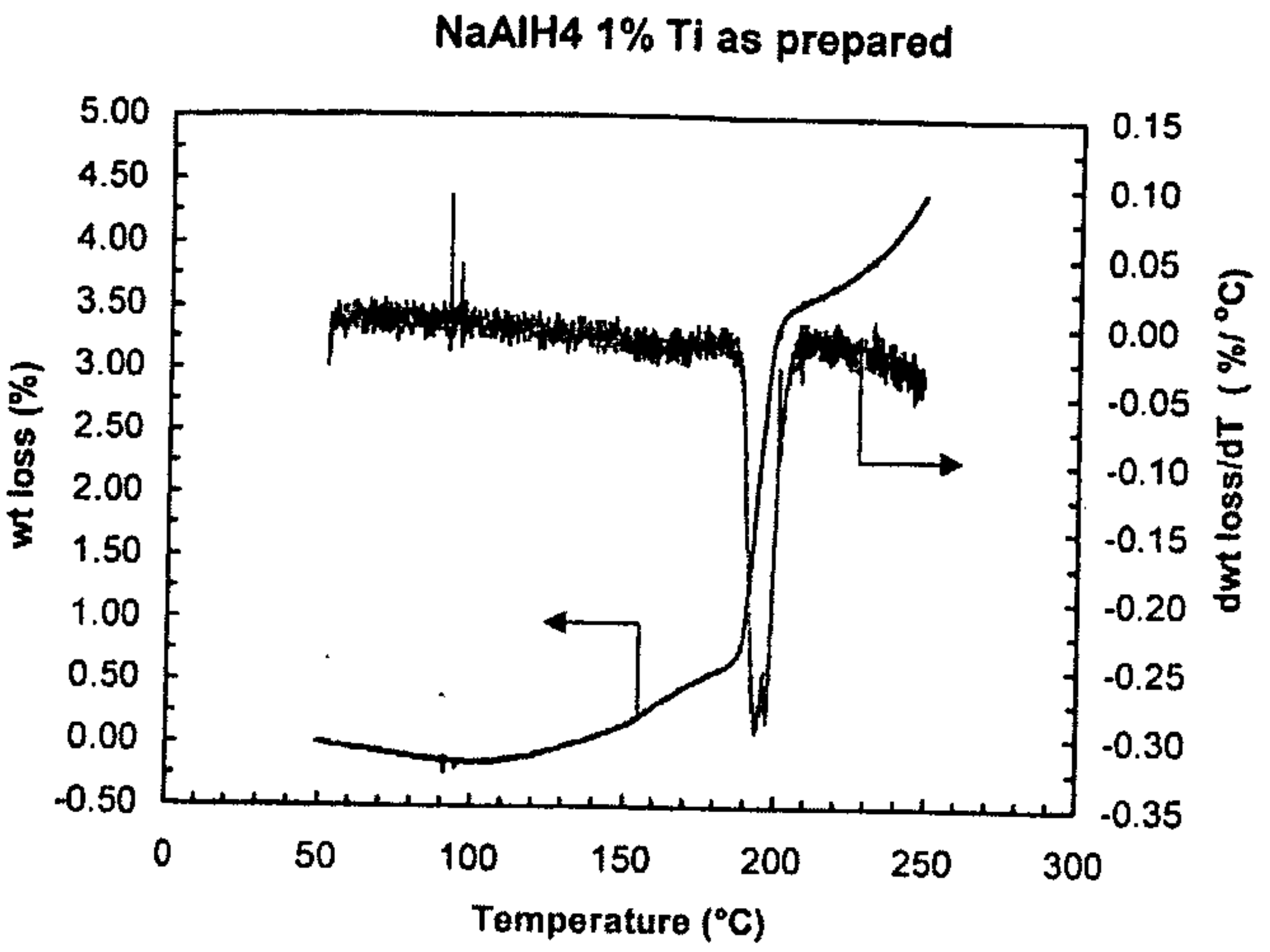


Figure 1

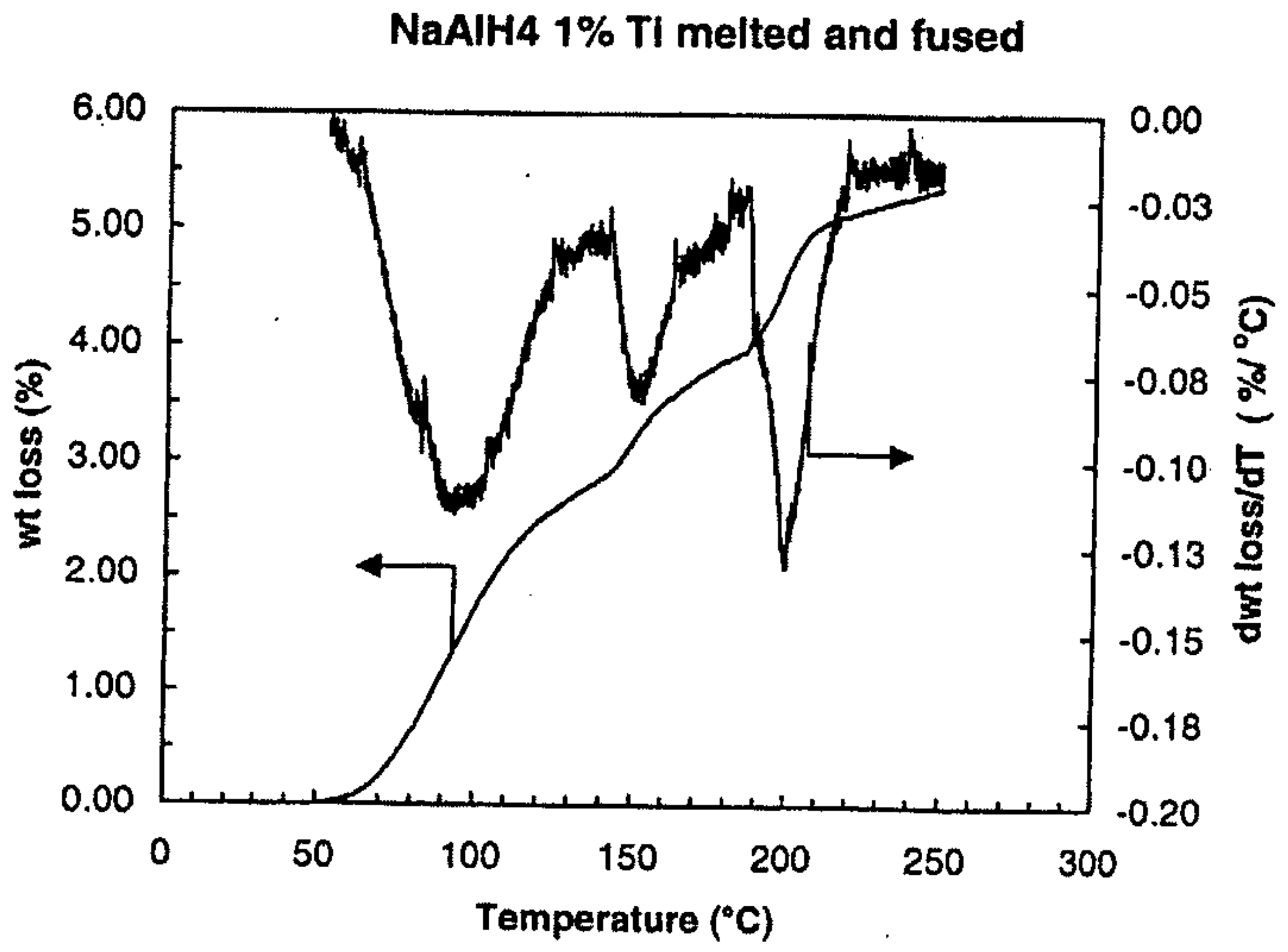


Figure 2

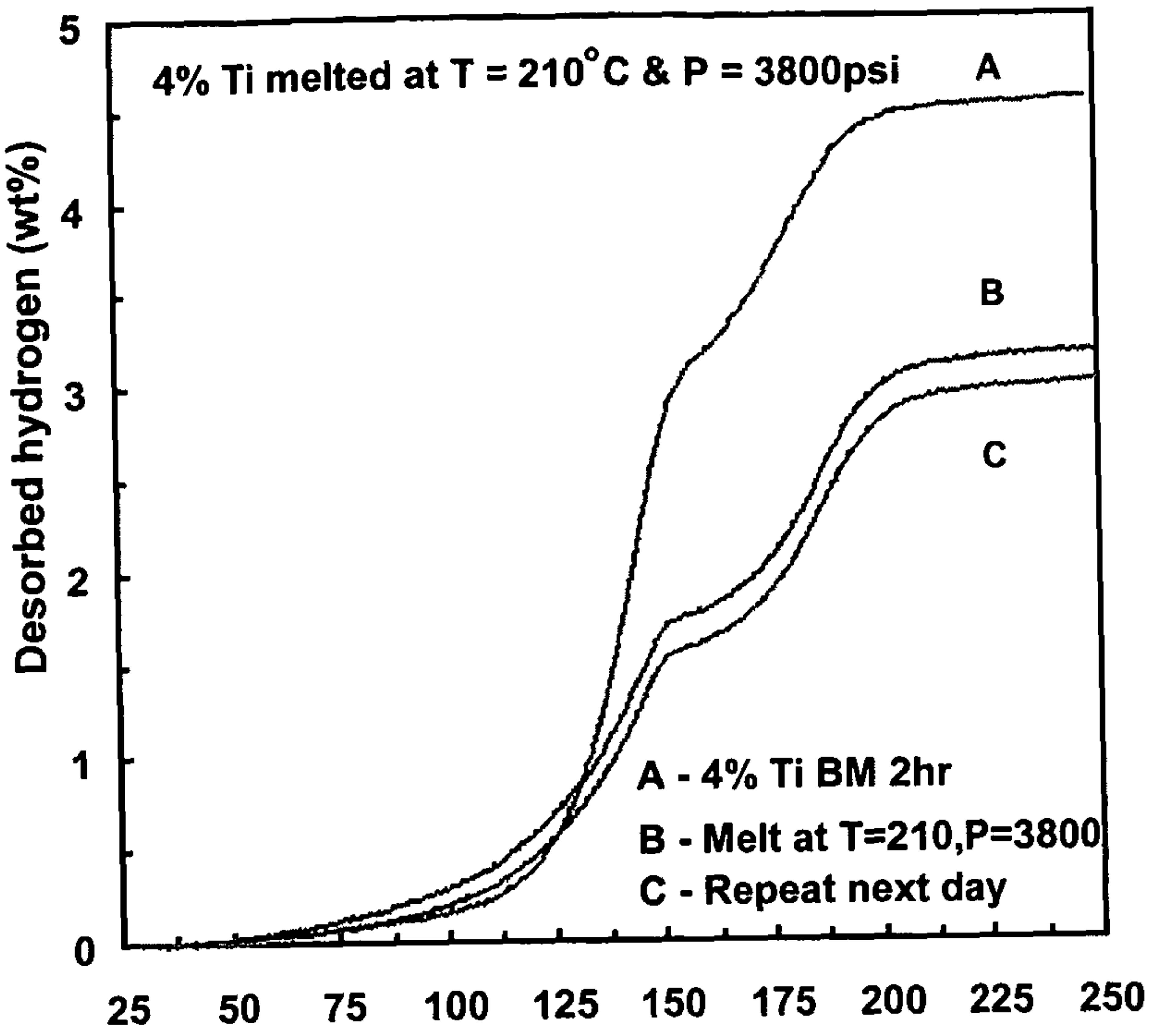

4% Ti melted at T = 210°C & P = 3800psi
A
B
C
A - 4% Ti BM 2hr
B - Melt at T=210,P=3800
C - Repeat next day
Desorbed hydrogen (wt%)
0
1
2
3
4
5
25
50
75
100
125
150
175
200
225
250
Temperature (°C)

Figure 3A

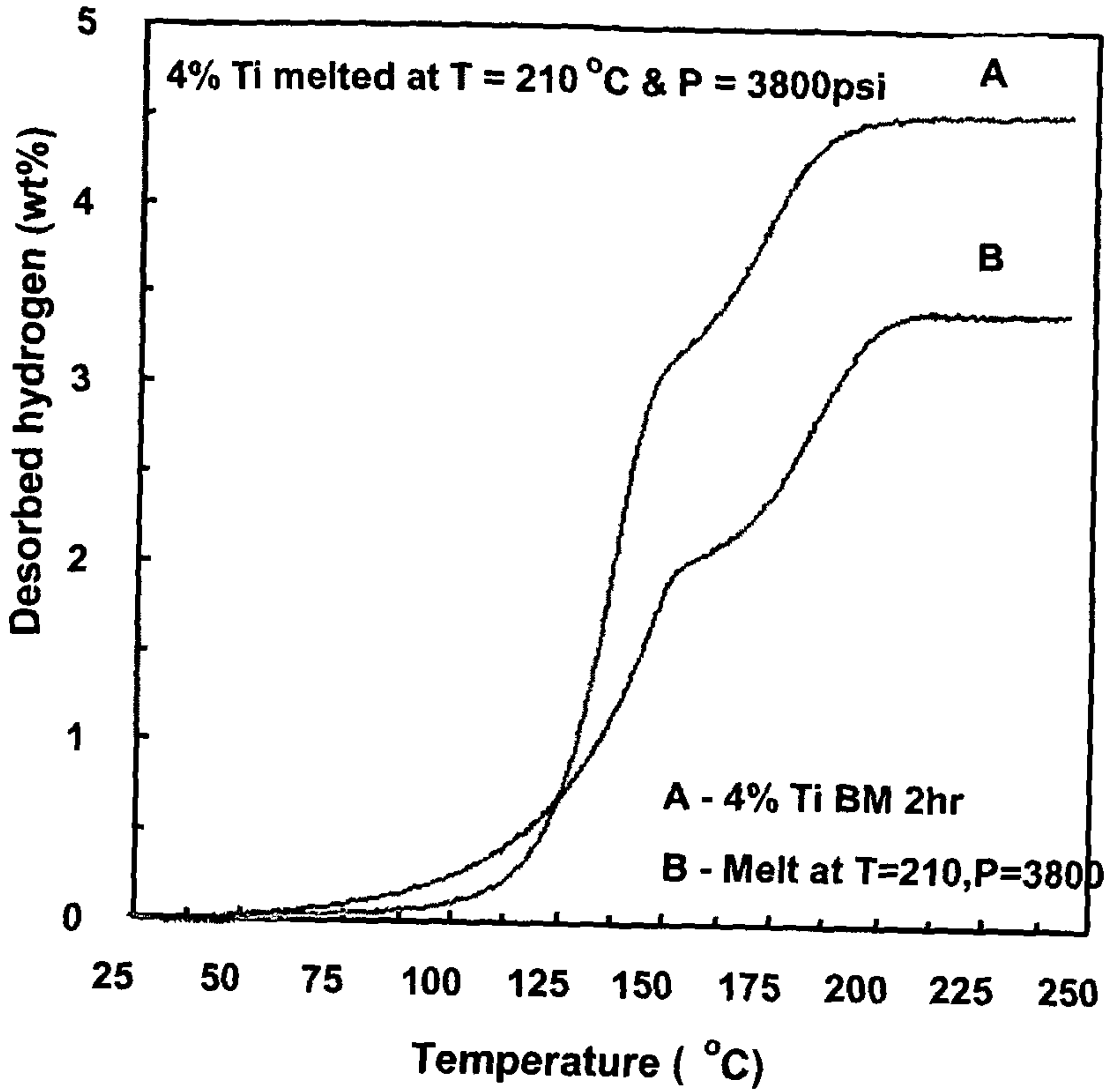

4% Ti melted at T = 210 °C & P = 3800psi
A
B
A - 4% Ti BM 2hr
B - Melt at T=210,P=3800
Desorbed hydrogen (wt%)
0
1
2
3
4
5
25
50
75
100
125
150
175
200
225
250
Temperature (°C)

Figure 3B

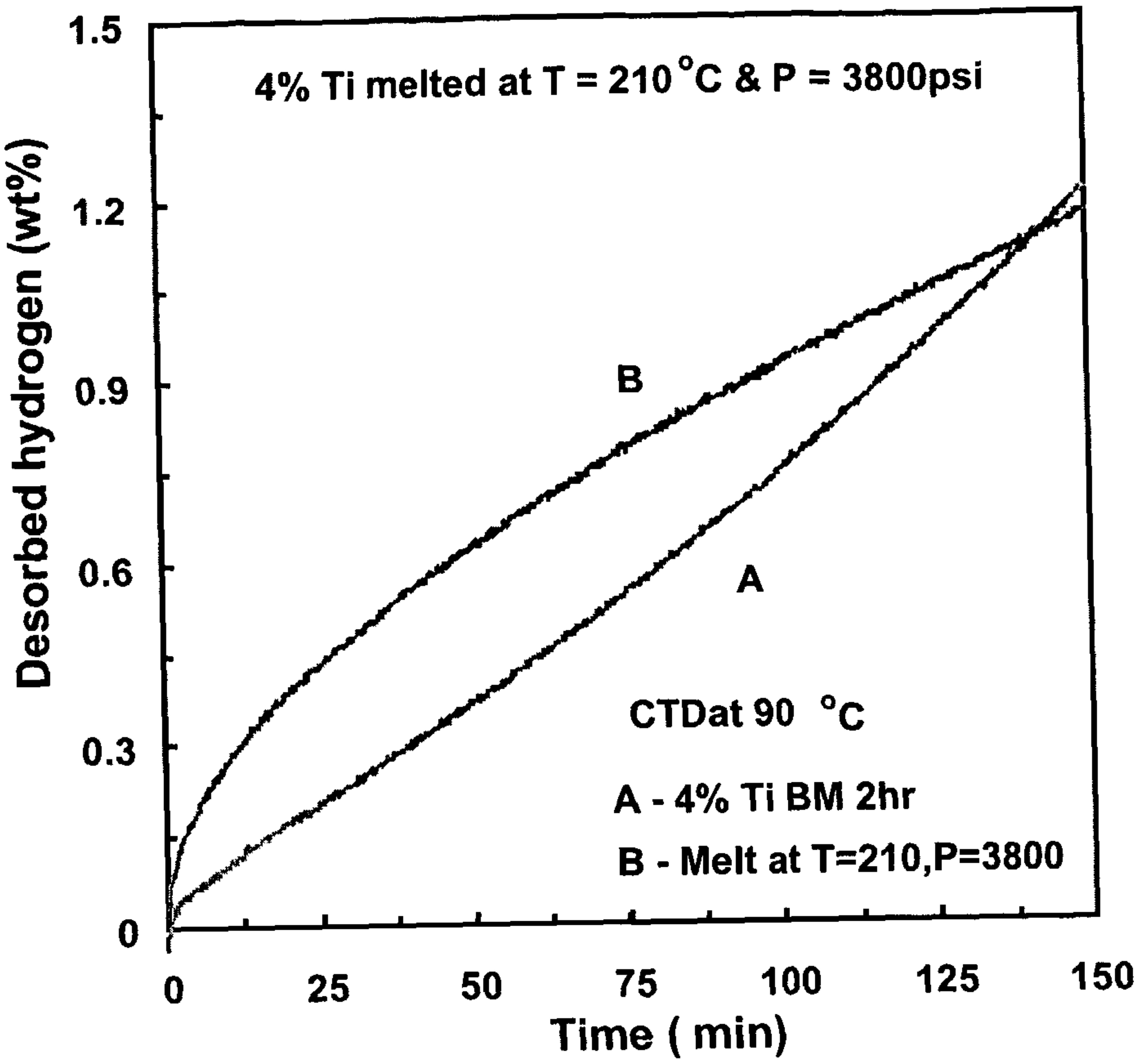

4% Ti melted at T = 210 °C & P = 3800psi
Desorbed hydrogen (wt%)
1.5
1.2
0.9
0.6
0.3
0
B
A
CTDat 90 °C
A - 4% Ti BM 2hr
B - Melt at T=210,P=3800
0
25
50
75
100
125
150
Time (min)

Figure 3C

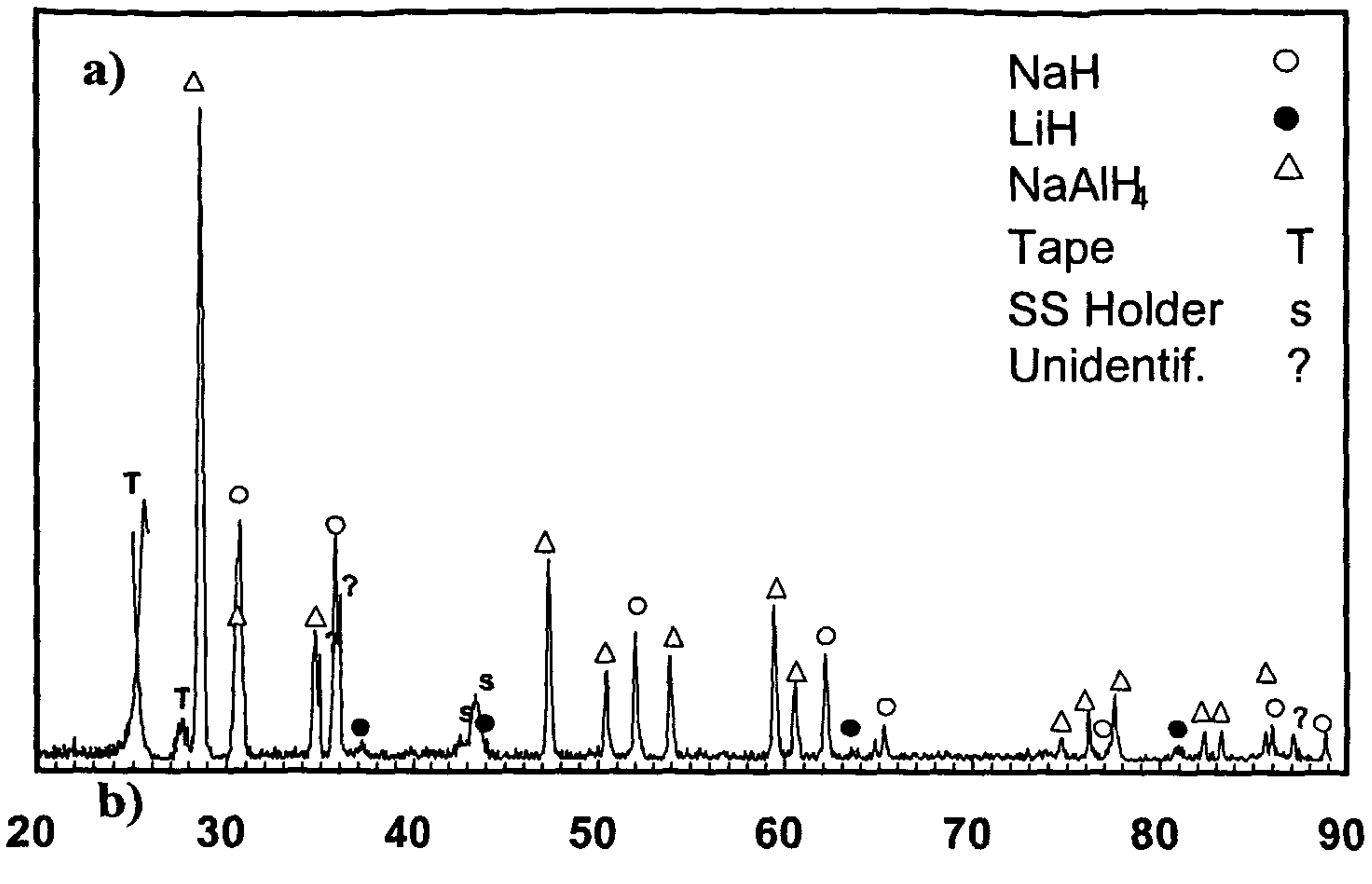


Figure 4A

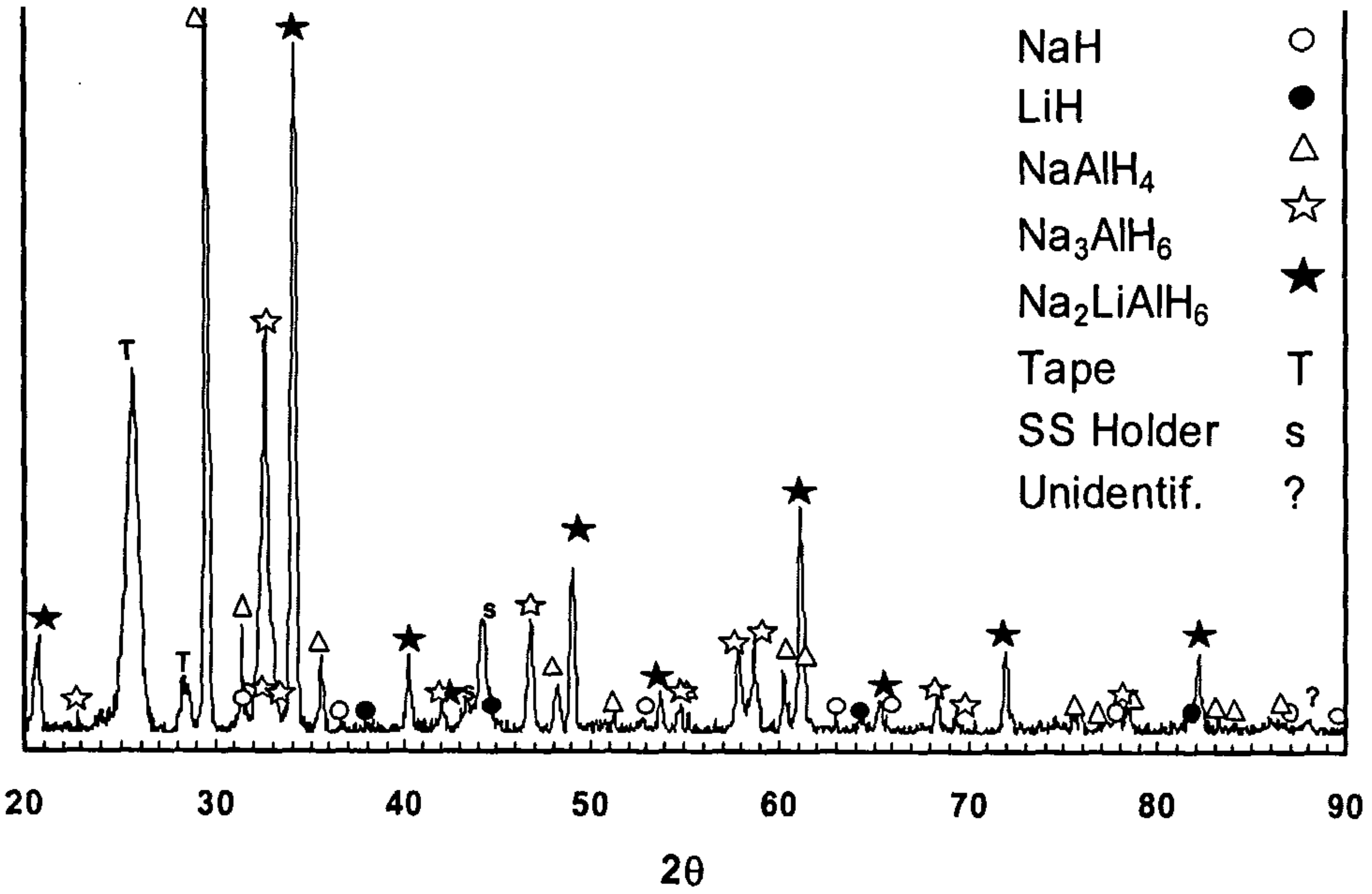
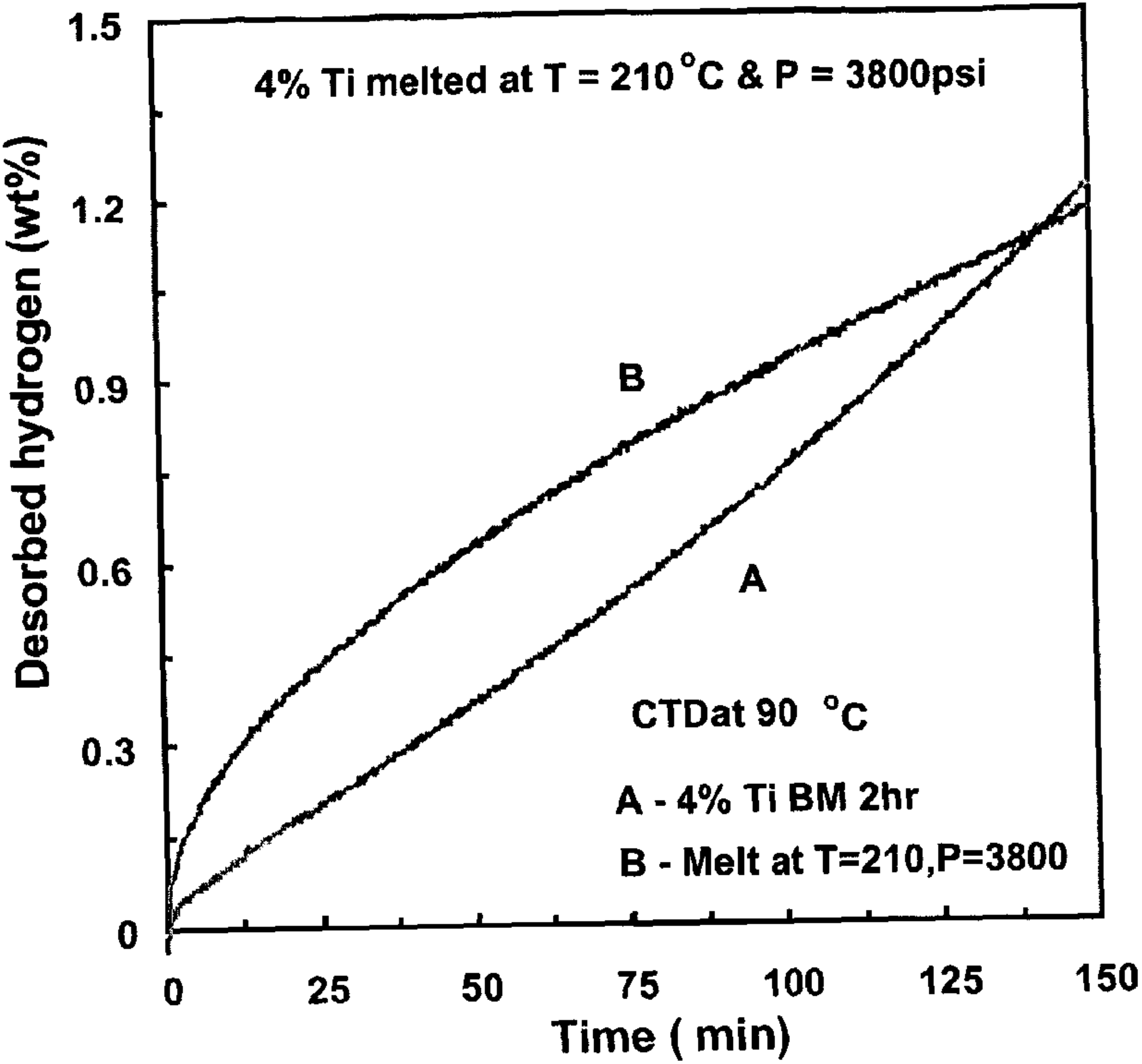


Figure 4B


4% Ti melted at T = 210 °C & P = 3800psi
Desorbed hydrogen (wt%)
1.5
1.2
0.9
0.6
0.3
0
B
A
CTDat 90 °C
A - 4% Ti BM 2hr
B - Melt at T=210,P=3800
0
25
50
75
100
125
150
Time (min)