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Zhang(10) **Pub. No.: US 2010/0064852 A1**(43) **Pub. Date: Mar. 18, 2010**(54) **METHOD FOR PURIFICATION OF METAL
BASED ALLOY AND INTERMETALLIC
POWDERS**(30) **Foreign Application Priority Data**

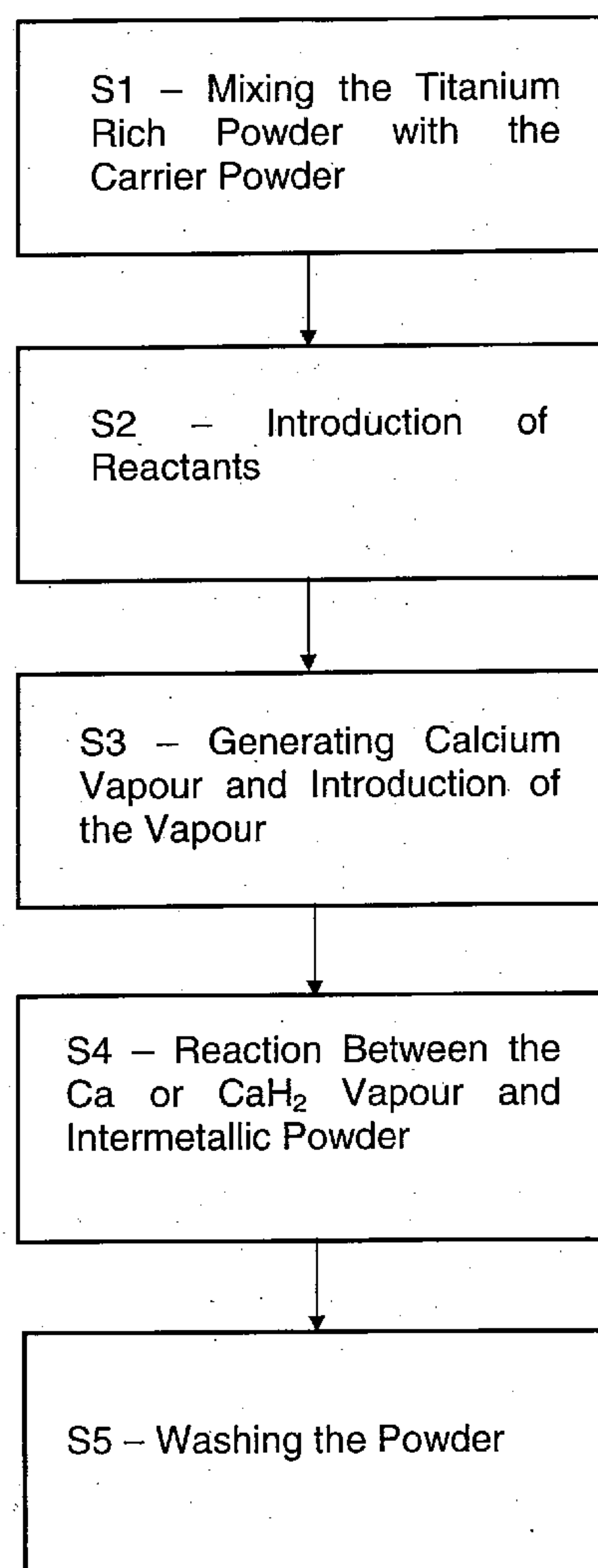
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(NZ)(57) **ABSTRACT**(21) Appl. No.: **12/517,016**(22) PCT Filed: **Nov. 30, 2007**(86) PCT No.: **PCT/NZ07/00351**§ 371 (c)(1),
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This invention relates to a method for purifying metal alloy and intermetallic powders. Particularly, the present invention relates to a method for the reduction or elimination of the content of the dissolved oxygen and to remove the metal oxide inclusions from metal alloy and intermetallic powders including the steps of: a) placing the metal in powder form into a reaction apparatus; b) introducing a suitable carrier substance to the metal powder; and c) introducing calcium vapour into the reaction apparatus to create a reaction between the metallic powder and calcium vapour thereby removing inclusions in the metal as shown in FIG. 11.



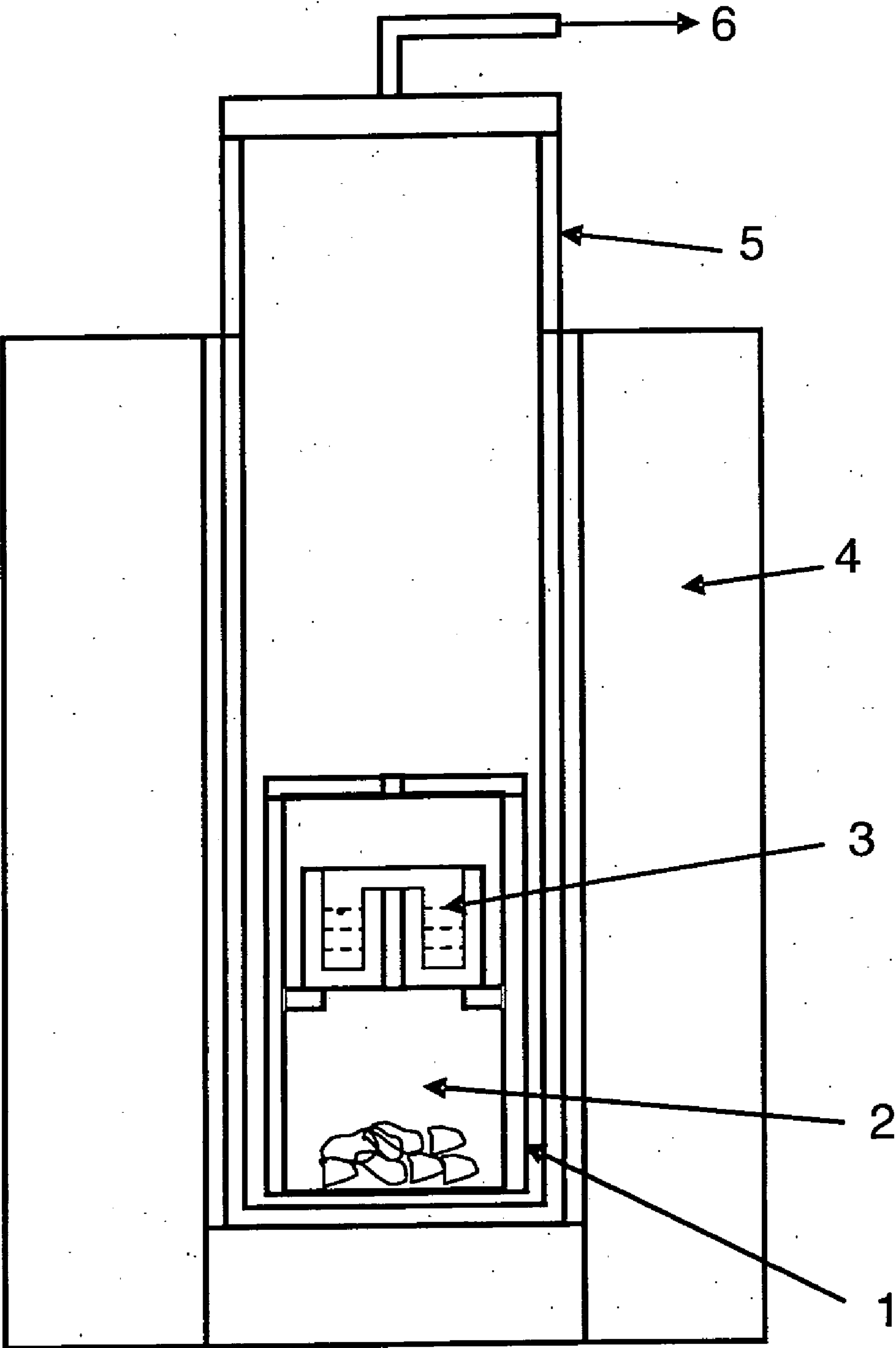


Figure 1(a)

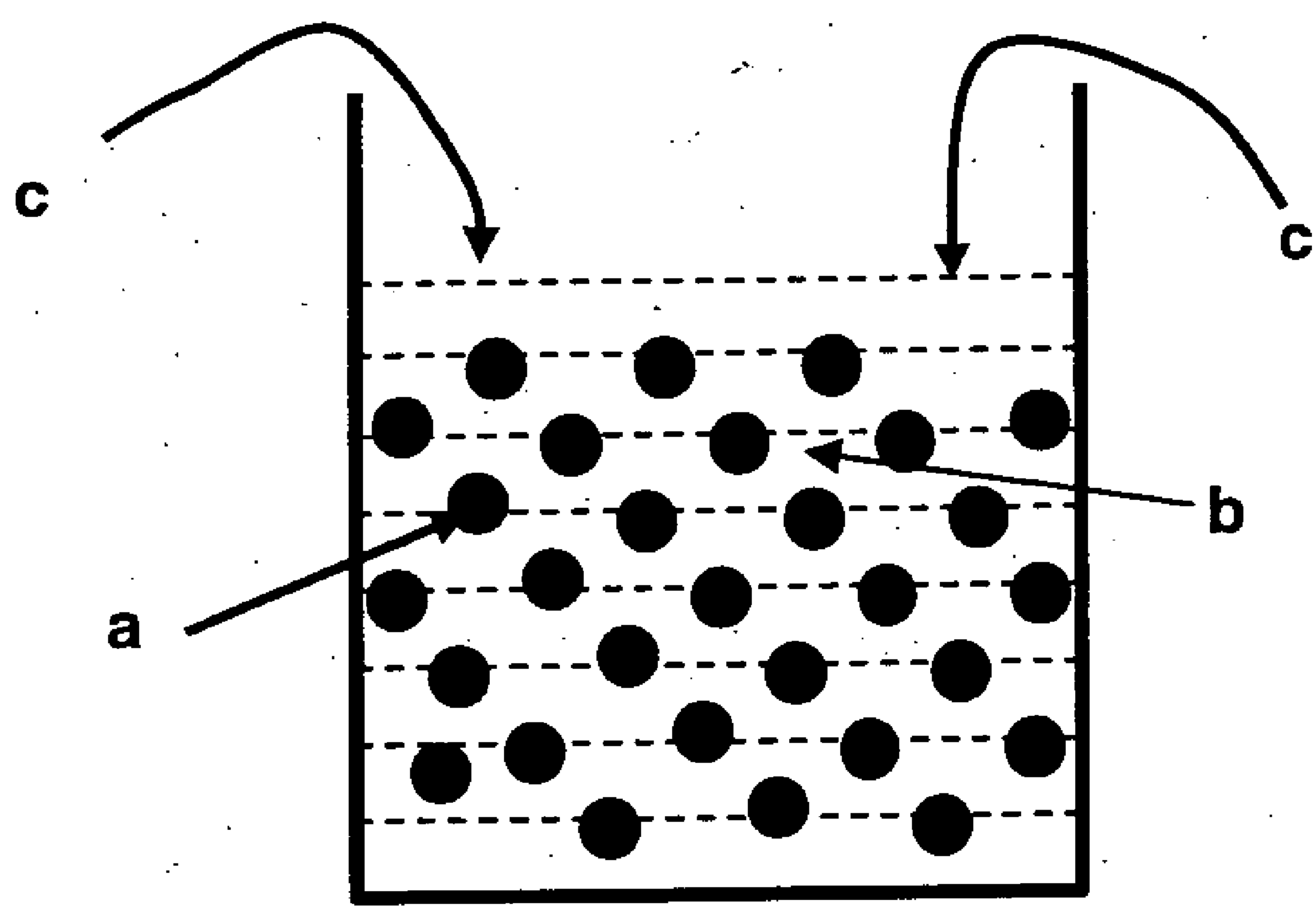


Figure 1(b)

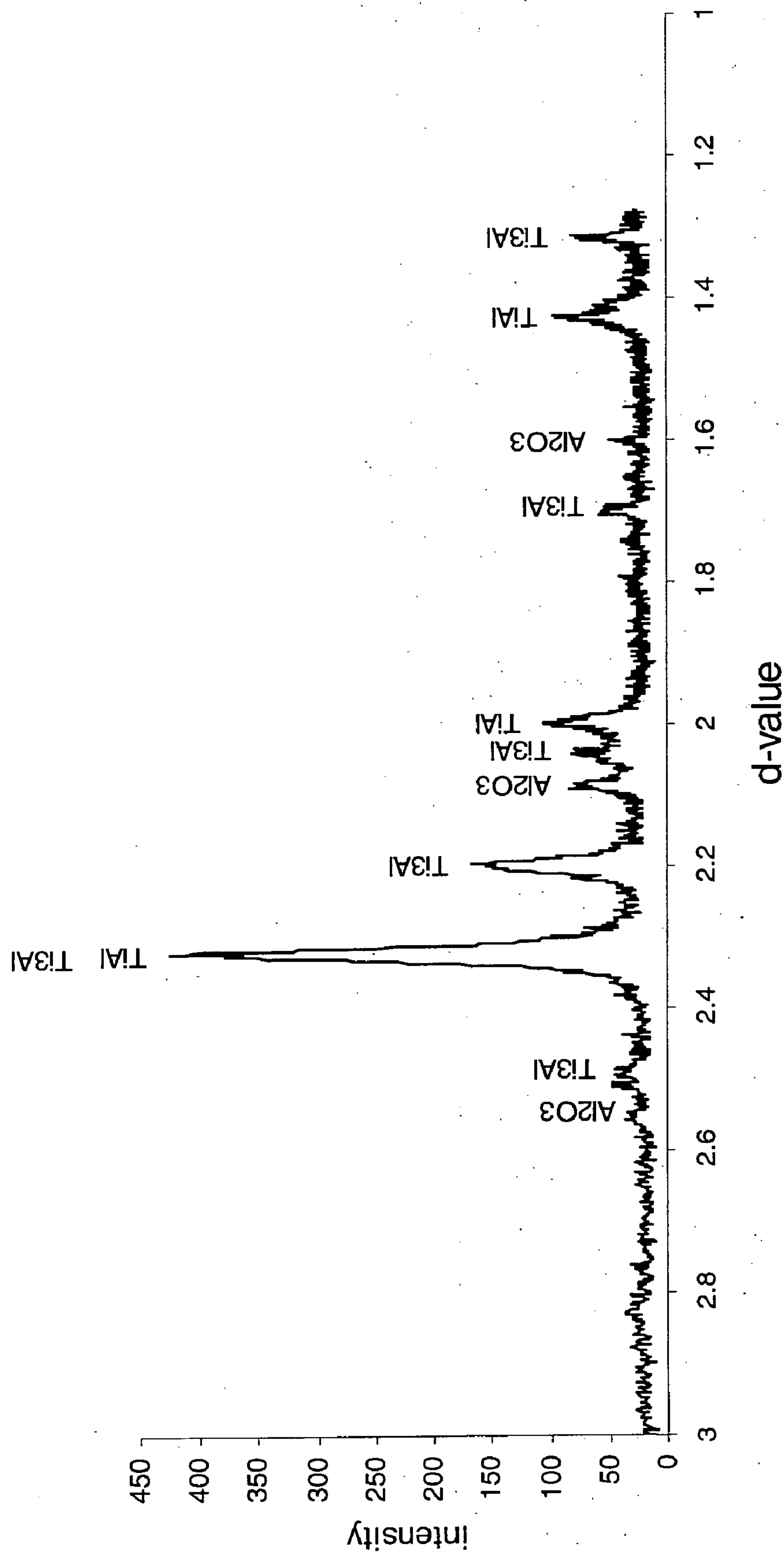


Figure 2

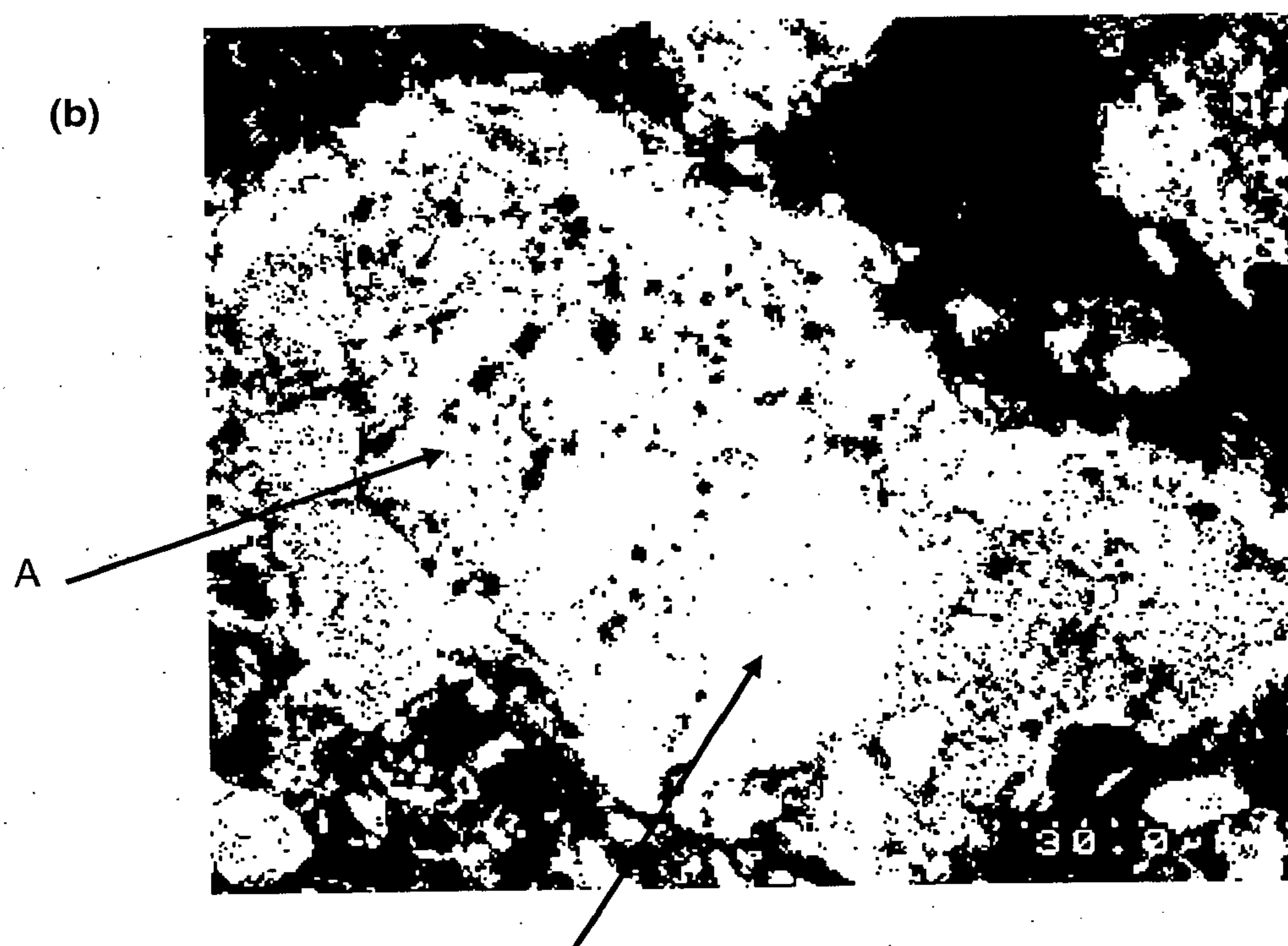
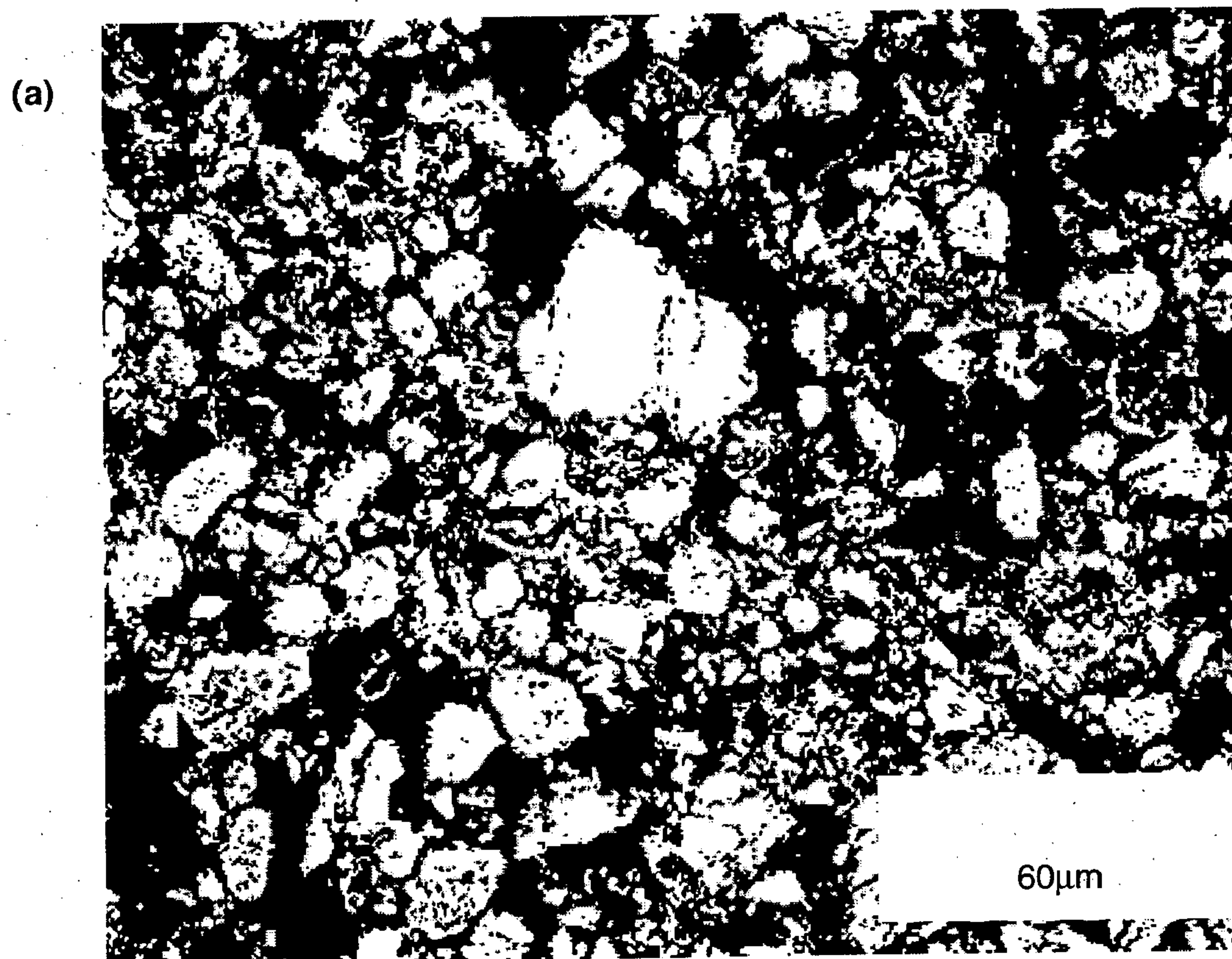


Figure 3

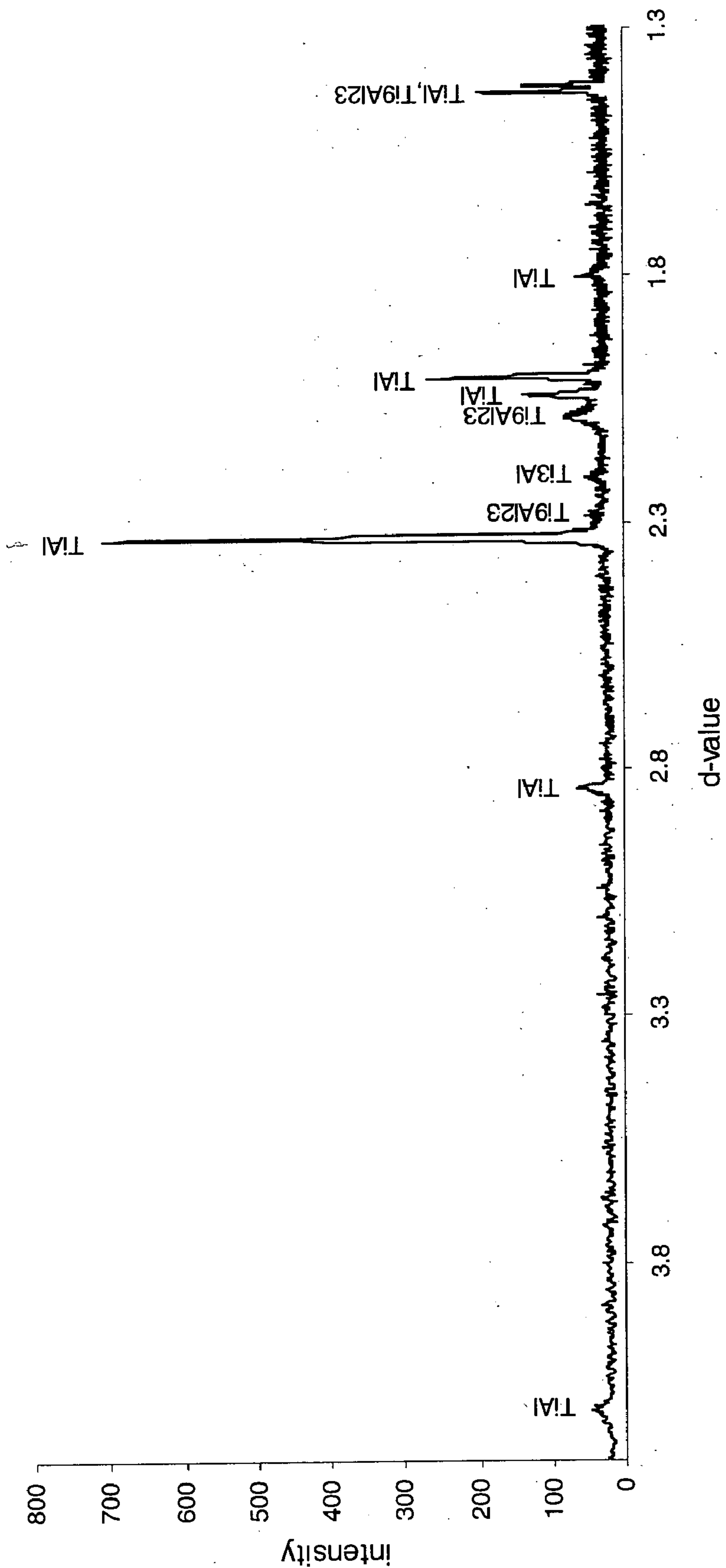
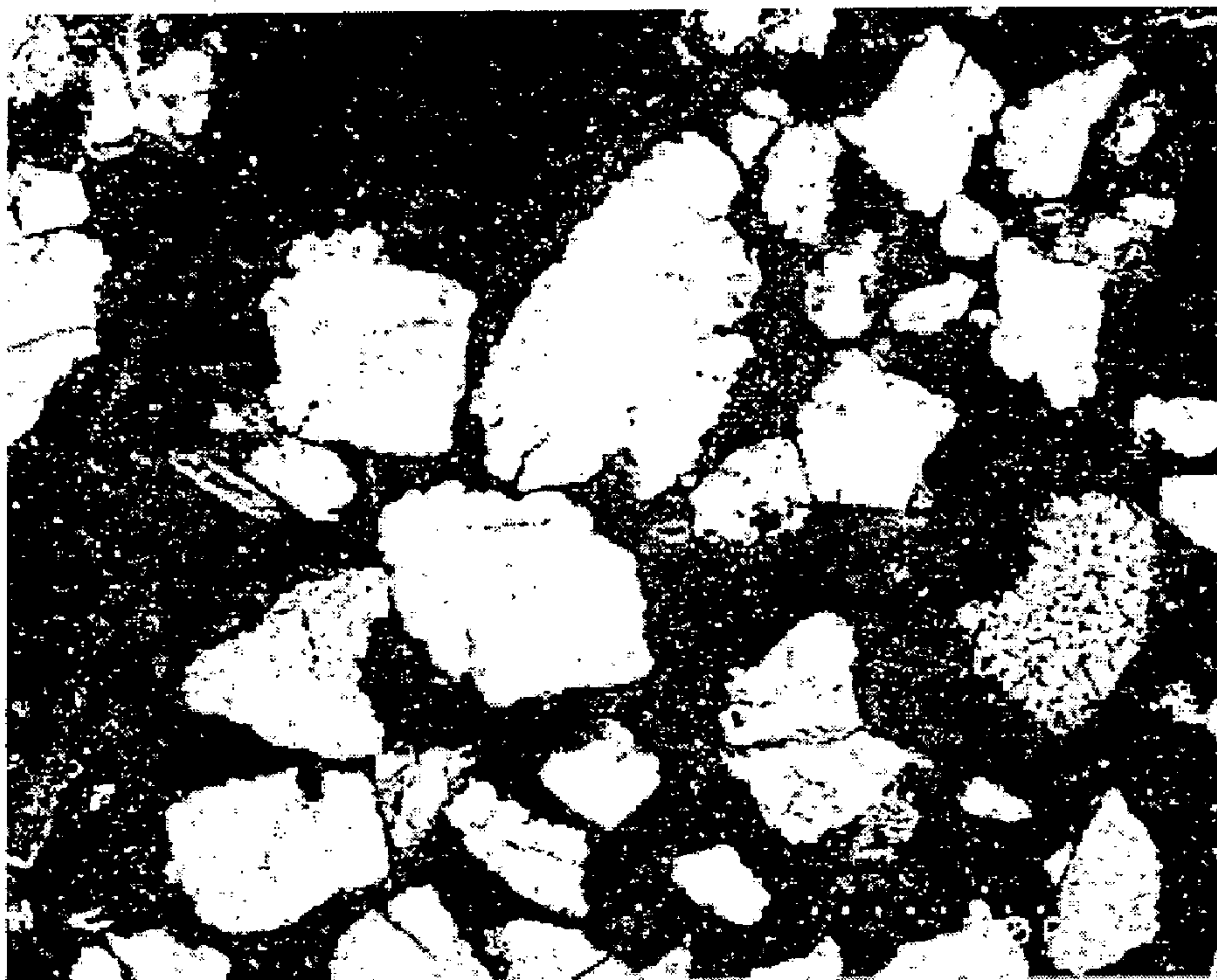


Figure 4

(a)



(b)



Figure 5

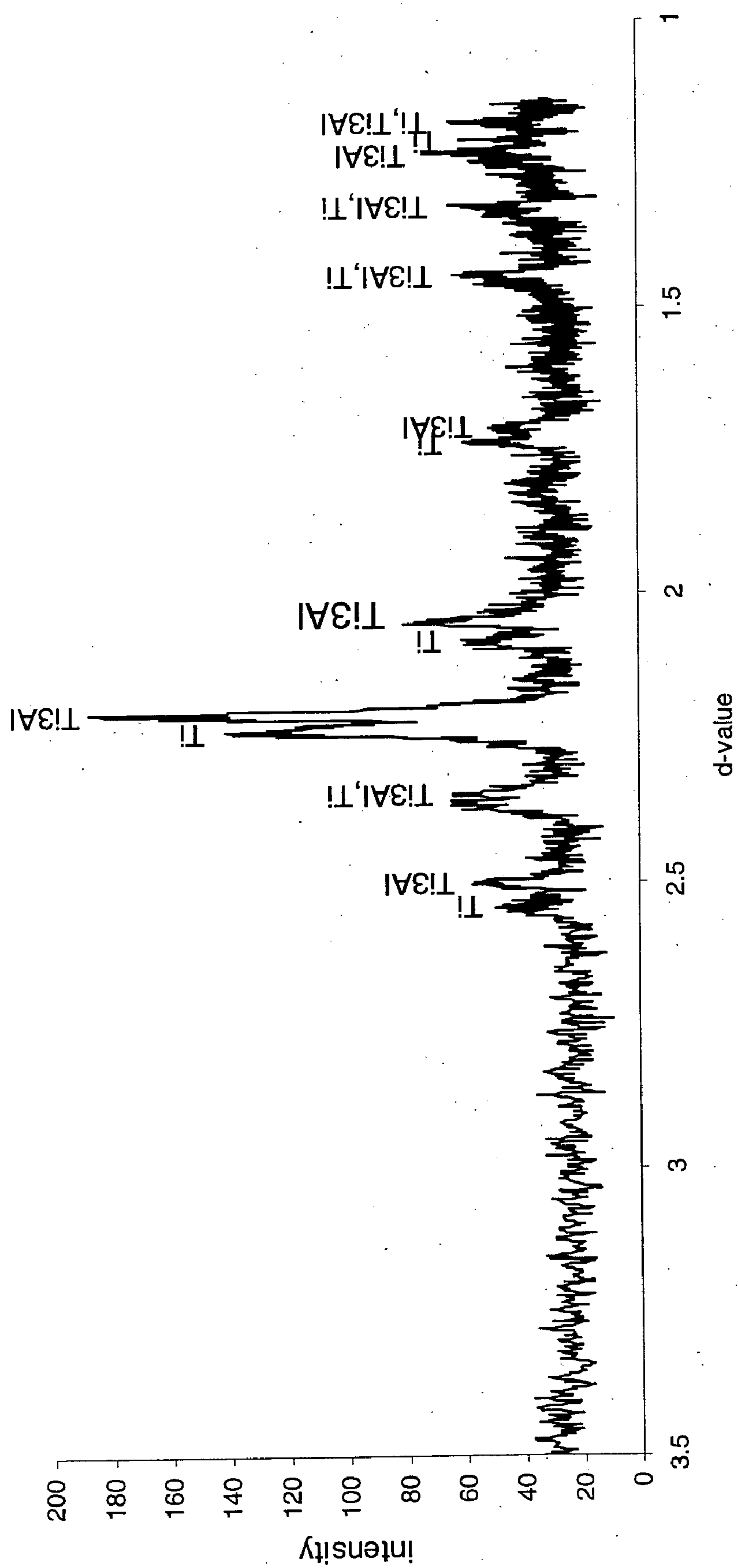


Figure 6

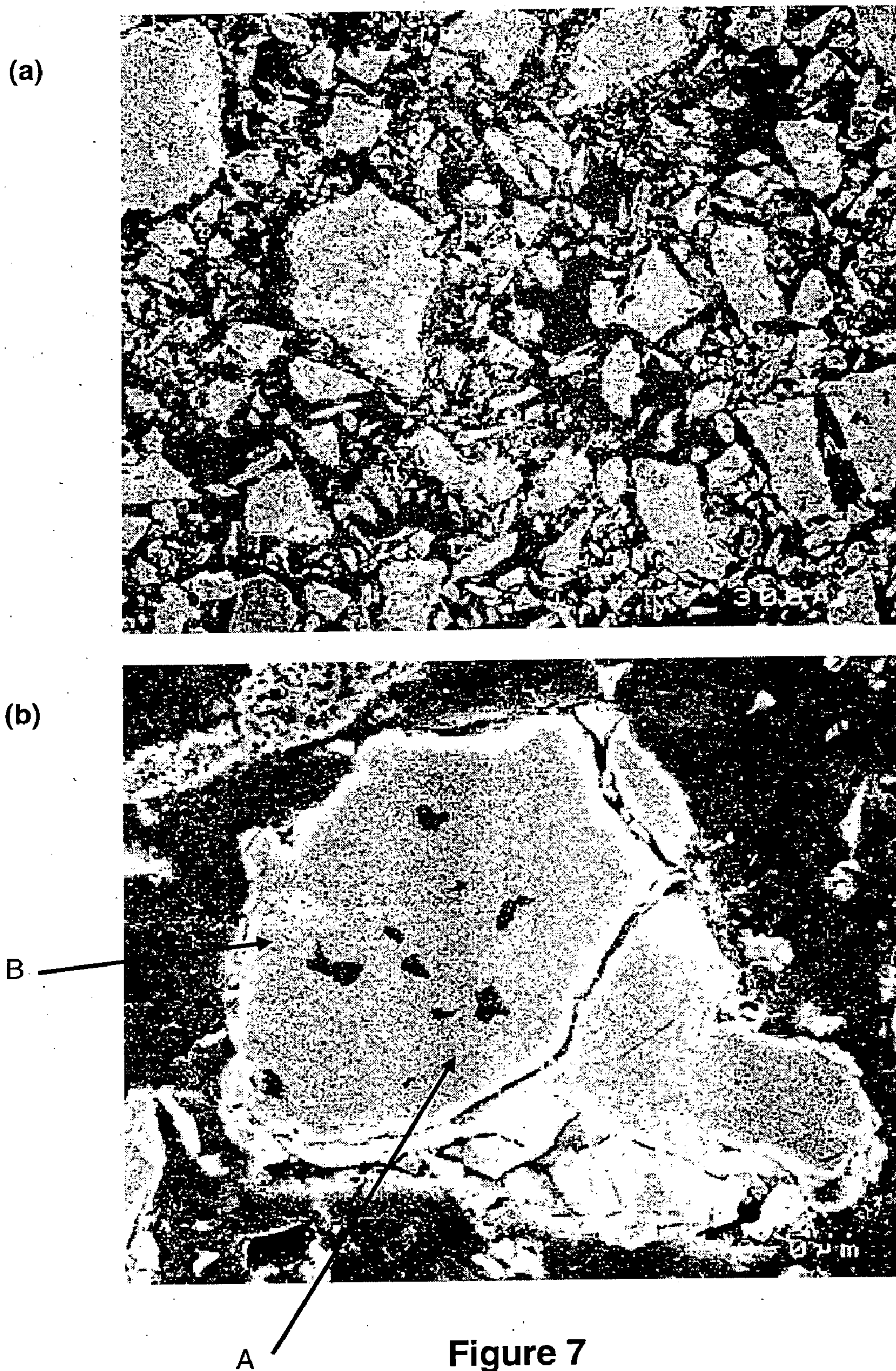


Figure 7

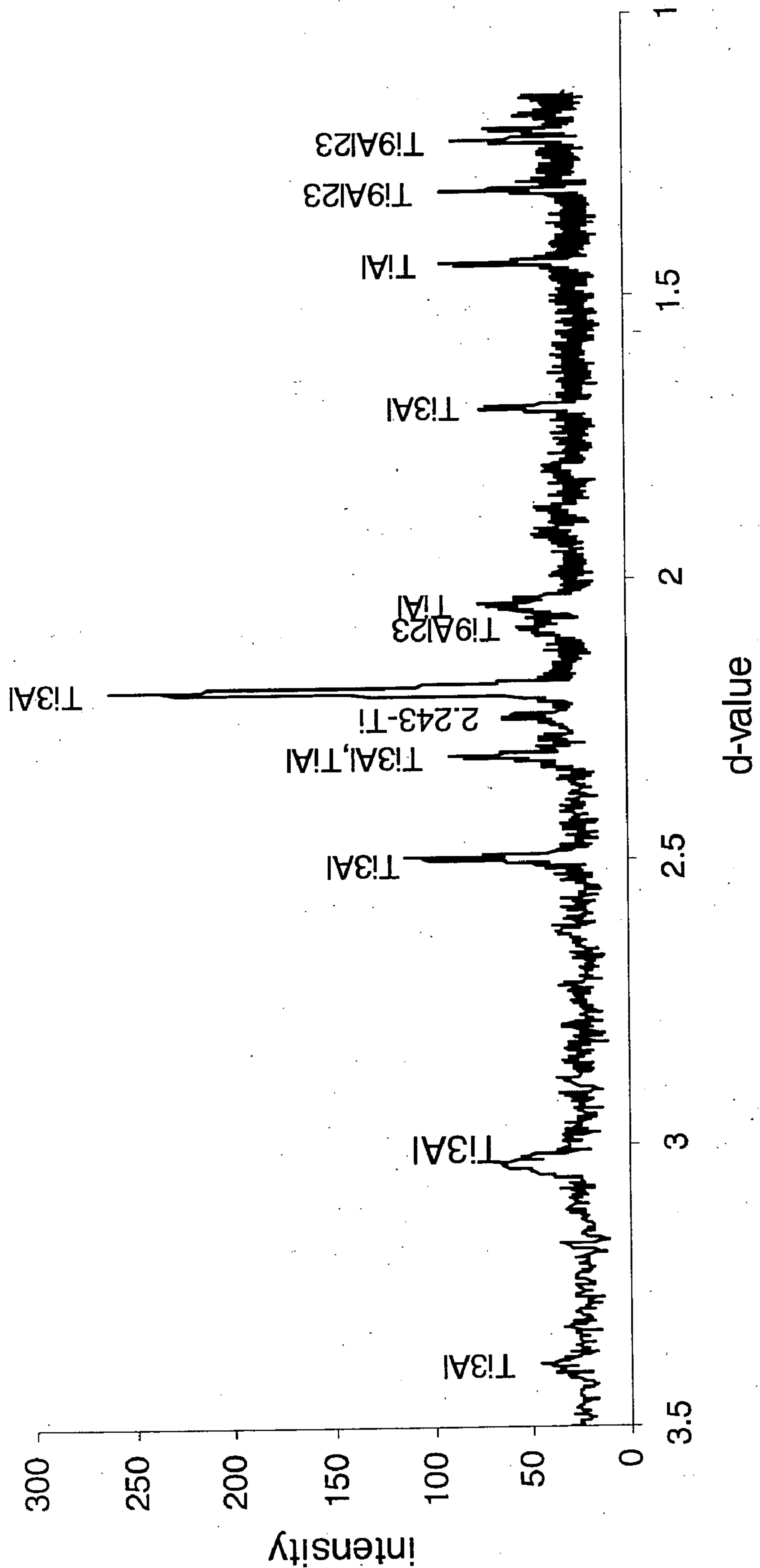


Figure 8

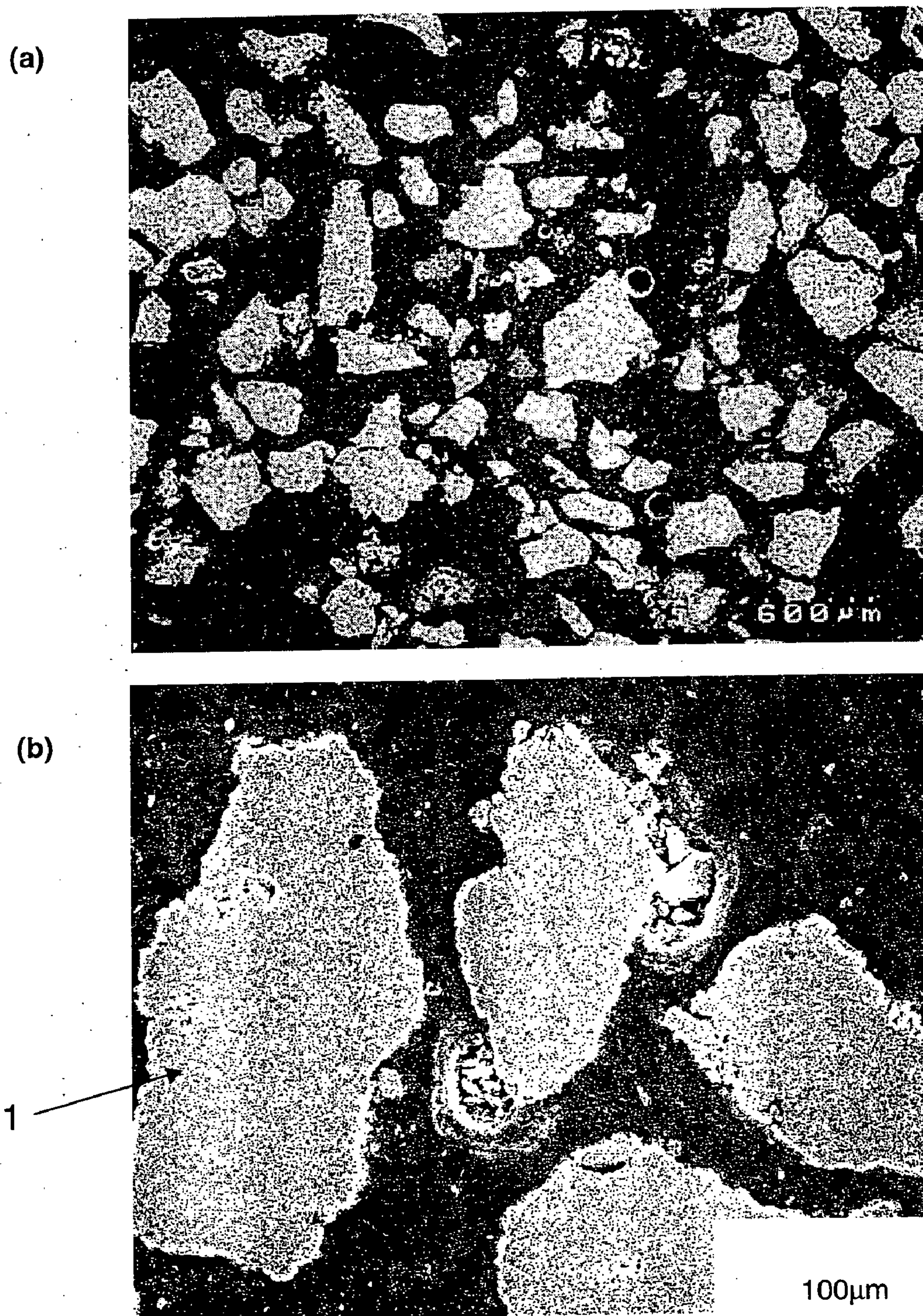


Figure 9

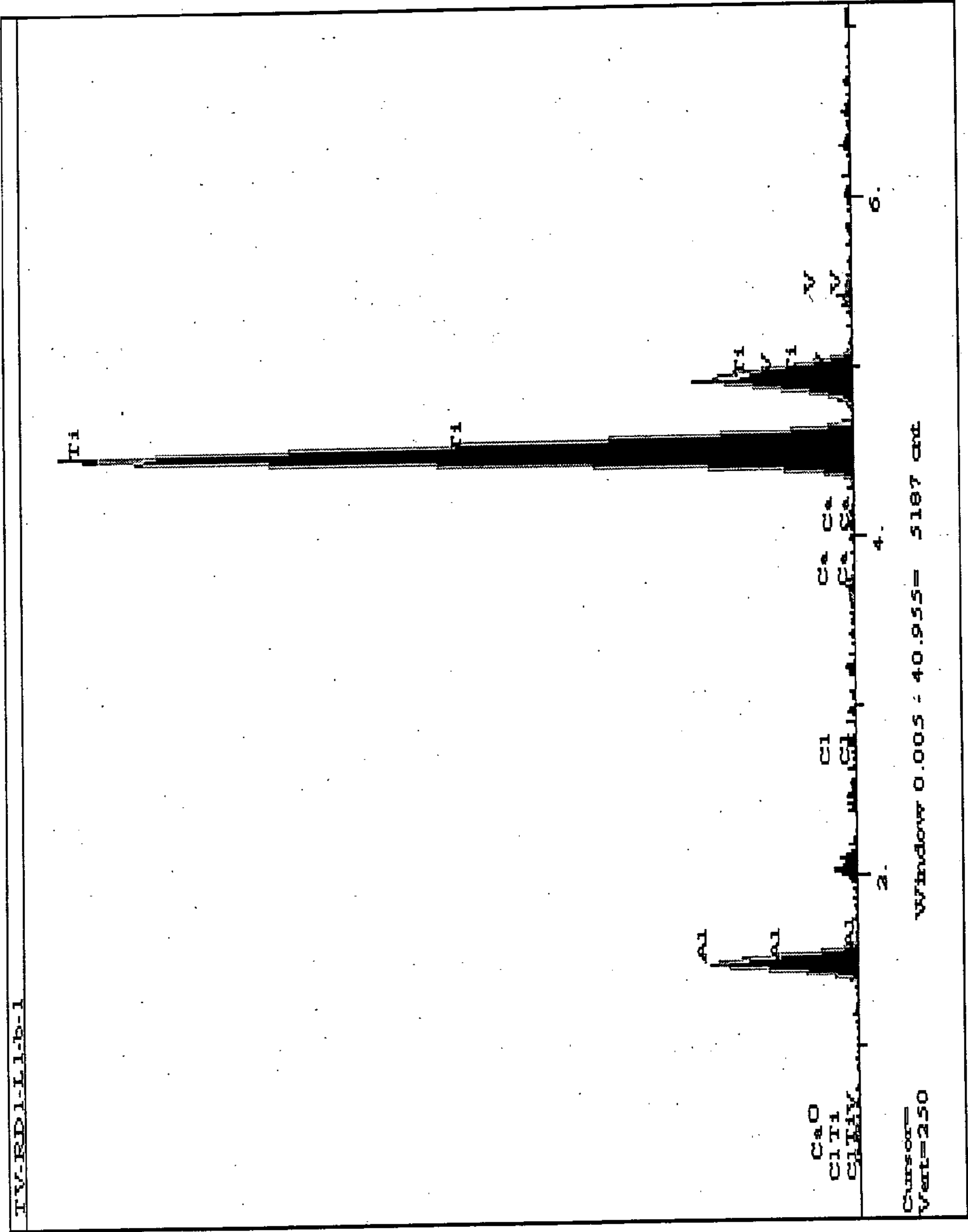
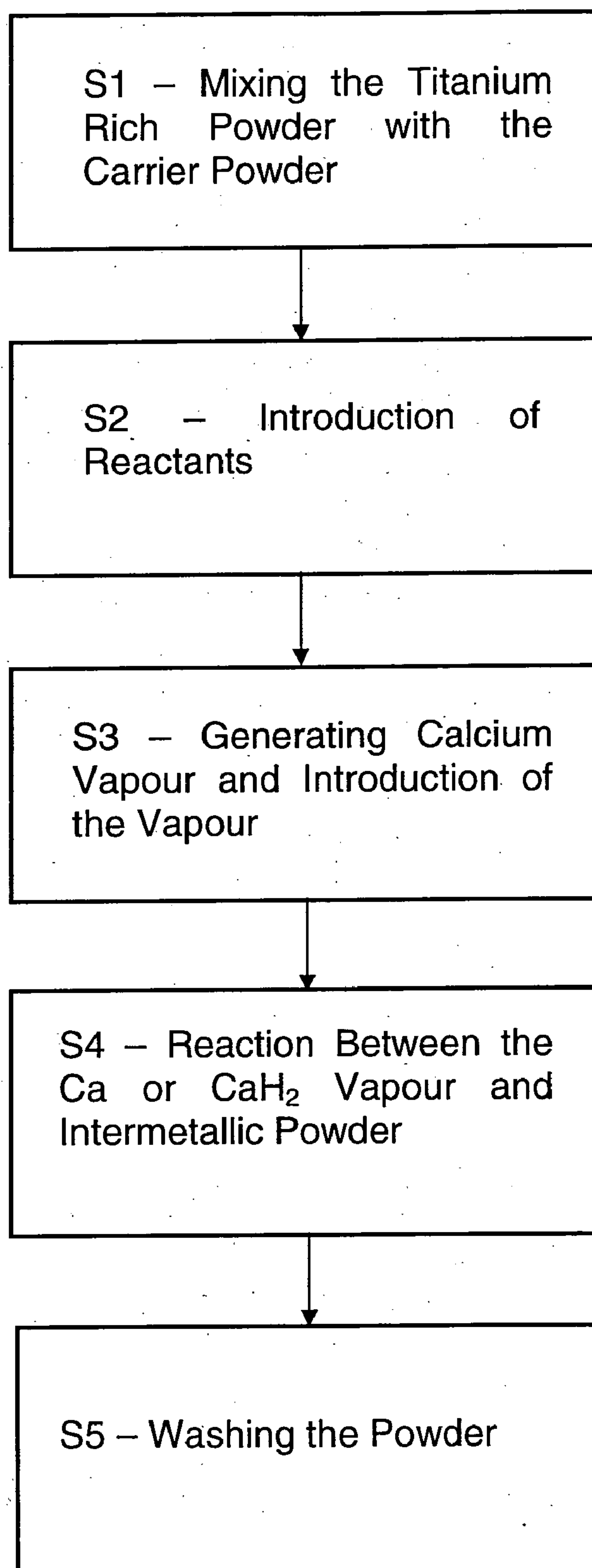


Figure 10(a)

Element	Atomic %	Weight %
O	0.000	0.000
Al	19.419	11.930
Cl	0.000	0.000
Ca	0.396	0.361
Ti	75.636	82.433
V	4.549	5.276
	100.000	100.000

Figure 10(b)

**Figure 11**

METHOD FOR PURIFICATION OF METAL BASED ALLOY AND INTERMETALLIC POWDERS

TECHNICAL FIELD

[0001] This invention relates to a method for purifying metal alloy and intermetallic powders. Particularly, although not exclusively the present invention relates to a method for the purifying titanium based alloy and intermetallic powders.

BACKGROUND ART

[0002] Metal, alloy and intermetallic powders produced by different processes such as liquid atomisation, mechanical milling, separation processes or a process that combines the above processes often contain a high content of dissolved oxygen (up to 10 wt % and metal oxide inclusions such as Al_2O_3). Such high content of dissolved oxygen and metal oxide inclusions can severely limit the range of practical applications of the metal, alloy and intermetallic powders because the high oxygen content and oxide inclusions can deteriorate the mechanical and other properties of the articles to an unacceptable level.

[0003] Therefore in many situations, it is essential to further purify the metal, alloy and intermetallic powders to significantly reduce the content of the dissolved oxygen and metal oxide inclusions.

[0004] Several methods have been developed that can be used to purify metal, alloy and intermetallic powders to reduce the oxygen content and/or metal oxide of the powders. For example, some methods make use of hydrogen and/or carbon as a reductant to extract the oxygen from the metal, alloy and intermetallic powders. However, the disadvantage of these methods is that they can not be applied to an active metal (such as titanium) based metal, alloy and intermetallic powders. This is due to thermodynamics where hydrogen and carbon are not sufficiently capable of extracting dissolved oxygen or reducing some of the metal oxide inclusions such as Al_2O_3 . For active metal based metal, alloy and intermetallic powders, more active metals such as calcium are often used as reductants to extract dissolved oxygen and reduce the metal oxide inclusions.

[0005] This is a well known art in the field of metallurgy. However, the methods to improve and establish the reaction and contact between the metal, alloy and intermetallic powder particles and the reductant metal are the subject of ongoing investigations.

[0006] For example, U.S. Pat. No. 2,834,667 discloses a process that uses calcium dispersed in a fused calcium chloride bath maintained at about 1000°C . or higher to deoxidise titanium scrap metal. This process can potentially be used to purify or deoxidise metal, alloy and intermetallic powders.

[0007] U.S. Pat. No. 4,923,531 discloses a process for deoxidation of titanium and similar metals which contain less than 1 wt % oxygen using a deoxidant in a molten metal carrier. The process described includes heating a liquid metal deoxidant such as calcium contained in a liquid metal carrier such as sodium to treat the oxidised metal. After removing the carrier and cooling, the metal is leached to result in a treated metal with lower residual oxygen. It has been shown that this process can be used to deoxidise titanium, titanium alloy and titanium based intermetallic powders.

[0008] However, the above mentioned processes or a similar type have limitations when they are used for deoxidising

metal, alloy and intermetallic powders, especially for those powders with fine particles of 100 micrometres or less in diameter. Since these processes involve mixing the deoxidant directly with the powder particles, the deoxidant must be free of any inclusions which cannot be leached by water or other suitable solutions such as mild acidic solutions that would not cause re-oxidation of the metal. This limitation prevents the use of low grade and low cost deoxidants, and thus can make the cost of the deoxidation process too high for many applications.

[0009] U.S. Pat. No. 6,171,363 discloses a method for producing tantalum/niobium metal powders by the reduction of their oxides with gaseous magnesium. In this process, metal powder from the group Ta, Nb, Ti, Mo, W, V, Zr, Hf preferably Ta or Nb, is made in a fine powder form by reduction of metal oxide by contact with a gaseous reducing agent, preferably an alkaline earth metal, to near complete reduction, leaching, further deoxidation and agglomeration. The powders produced using this method are sinterable to capacitor anode form and processable to other usages.

[0010] However, the disadvantage of this method is that the particles of the powder to be reduced or deoxidised are packed in a bed. Therefore, the effectiveness for the contact between the gaseous reductant and the powder particles to be reduced is limited because of the exposed surface area of each of the particles is reduced by the physical contacts with those particles surrounding it. With high powder packing density which is often associated with fine particle sizes, the exposed surface area can be significantly limited and thus reduce the reduction effectiveness to a level that lead to impractical reaction times in order to achieve complete reduction or deoxidation.

[0011] Furthermore, in this method; the powder bed is required to be heated to a sufficiently high temperature in order to achieve a fast enough reaction or deoxidation rate. At this high temperature, metal, alloy and intermetallic powders are often partially sintered during the reduction or deoxidation process, and the sintering creates physical links among the particles. Although these links can be broken by subsequent milling, it increases the costs of the process. For some metal and alloy powders such as titanium alloys, the milling is often a difficult process because of their high ductility. In addition, the milling process changes the size and morphology of the original powder particles, and this is not considered a desirable property.

[0012] It would be an advantage of the present state of the art to have some general method which can be used to lead to significantly reduce the content of the dissolved oxygen and to remove the metal oxide inclusions from active metal based metal, alloy and intermetallic powders, and overcome the difficulties associated with other methods, especially those mentioned above as examples.

[0013] All references, including any patents or patent applications cited in this specification are hereby incorporated by reference. The discussion of the references states what their authors assert, and the applicants reserve the right to challenge the accuracy and pertinency of the cited documents. It will be clearly understood that, although a number of prior art publications are referred to herein, this reference does not constitute an admission that any of these documents form part of the common general knowledge in the art, in New Zealand or in any other country.

[0014] It is an object of the present invention to address the foregoing problems or at least to provide the public with a useful choice.

[0015] Further aspects and advantages of the present invention will become apparent from the ensuing description which is given by way of example only.

DISCLOSURE OF INVENTION

[0016] According to one aspect of the present invention there is provided a method of purifying a metal including the steps of:

[0017] a) placing the metal in powder form into a reaction apparatus;

[0018] b) introducing a suitable carrier substance to the metal powder; and

[0019] c) introducing calcium vapour into the reaction apparatus to create a reaction between the metallic powder and calcium vapour thereby removing inclusions in the metal.

[0020] Throughout the present specification the term 'metal' in accordance with the present invention should be understood to include metal based alloy, metallic or intermetallic material consisting of a mixture of at least two metals or of metallic with non-metallic elements. While it should be appreciated that there is at least two substances in a metal based alloy, there is theoretically no limit to the number of substances that make up a metal based alloy.

[0021] However, this should not be seen as a limitation on the embodiments envisaged for this invention. The metals that predominantly make up the metal based alloy can include nickel, platinum, aluminium, palladium and any others from the periodic table.

[0022] It should be appreciated to those skilled in the art that a metallic or intermetallic powder is a substance that contains one or more metal compounds divided into many small individual particles.

[0023] Preferably, the particles are less than 100 micrometres.

[0024] The term purification or grammatical variations in the specification should be understood to mean the reduction or elimination of the content of dissolved oxygen and metal oxide inclusions in a metal, alloy or intermetallic powder. This term may be used interchangeably with the term 'deoxidation' throughout the specification.

[0025] In preferred embodiments, the metal inclusions may be Al_2O_3 . However, this should not be seen as a limitation on the embodiments envisaged for this invention. Conceivably, any other oxygen containing compound inclusions such as CuO , V_2O_5 , Nb_2O_3 or Cr_2O_3 , V_2O_5 , Nb_2O_3 or Cr_2O_3 may also be removed.

[0026] In preferred embodiments, the metal, alloy and intermetallic powders requiring deoxidation may consist of titanium phase, titanium rich metallic phases such as $\text{Ti}(\text{Al})$ solid solution, and intermetallic compounds such as TiAl , and/or Ti_3Al containing dissolved oxygen and Al_2O_3 inclusion particles.

[0027] For ease of reference throughout the specification, TiAl and Ti_3Al will now be collectively referred to as Ti_xAl . This term should not be seen as limiting.

[0028] These titanium rich powders may be products manufactured from any method known in the prior art base that require further purification or 'deoxidation'.

[0029] An advantage of this method is that it is able to remove the oxide phases in the material and further reduce the

oxygen content and the titanium rich powder to below 0.4 weight % to produce high value titanium based metallic and intermetallic powders such as Ti_xAl powders.

[0030] Throughout the present specification the term 'suitable carrier substance' in accordance with the present invention should be understood to mean any substance that is solvable in water or mild acidic solutions, has a sufficiently low melting point and has a substantial liquid solubility of a metal deoxidant.

[0031] Preferably, a titanium rich metallic or intermetallic powder containing intermetallic compounds such as Ti_xAl and/or metallic phases such as $\text{Ti}(\text{Al})$ solution and Al_2O_3 particles is homogeneously mixed with a carrier substance such as calcium chloride (CaCl_2) or hydrated calcium chloride ($\text{CaCl}_2 \cdot \text{H}_2\text{O}$) placed into a reaction apparatus.

[0032] The design of the apparatus requires specific modifications to standard apparatus known in the industry for optimum reactivity and is shown in FIG. 1(a) including the following labelled componentry: Reaction container (1), chamber for holding Ca granules (2), Chamber for holding the mixture of metallic or intermetallic powder and carrier liquid (3), Furnace (4), Stainless steel retort (5) and connection to vacuum pump and argon supply (6).

[0033] Preferably, the reaction apparatus has two chambers enabling the metallic or intermetallic powder/carrier mixture to remain separate from the metal deoxidant such as Ca or CaH_2 .

[0034] In preferred embodiments the metal deoxidant may be Ca in the form of a solid. In one embodiment, the Ca solid may be granules.

[0035] However, this should not be seen as a limitation on the embodiments envisaged for this invention. The Ca solid may be in the form of a powder, crystals, fused solid or any other such solid.

[0036] Preferably, the $\text{Ti}_x\text{Al}/\text{CaCl}_2 \cdot \text{H}_2\text{O}$ powder mixture and Ca granules are kept in separate chambers of the apparatus and heated to a temperature in a typical range of 700-1400° C., and held at this temperature for a time period in a typical range of 1 minute to 100 hours to allow the generation of Ca vapour.

[0037] More preferably, the Ca vapour generated during heating is introduced to the other chamber where the mixture of the metallic or intermetallic powder and the carrier powder is kept and is heated to the same typical temperature range of 700-1400° C.

[0038] In preferred embodiments the temperature allows the carrier to melt, a substantial fraction of the metal deoxidant to turn into vapour and sufficiently high interactions between the metal, alloy or intermetallic powder particles and the deoxidant vapour to interact through the medium of the carrier.

[0039] Preferably, the interactions between Ca vapour and the Ti_xAl powder particles dispersed in the liquid carrier are achieved through the Ca atoms dissolved in the liquid medium.

[0040] Throughout the present specification the term 'introduce' in accordance with the present invention should be understood to mean delivering the Ca/CaH_2 vapour and carrier substance for reaction with the metallic or intermetallic powder.

[0041] It is envisaged a continuous flow of Ca vapour will be maintained throughout the reaction with an analysis of the reaction kinetics.

[0042] The reaction process utilises the Ca vapour to react with the titanium rich metallic or intermetallic powder (containing oxide particles and dissolved oxygen) to reduce the residual oxide particles in the powder and scavenging the dissolved oxygen, reducing the oxygen level in the metallic and intermetallic phases to below 0.4% in weight. The reaction will lead to the formation of CaO and $\text{CaO} \cdot \text{Al}_2\text{O}_3$ phases in particle form which may also be dissolved in the liquid carrier.

[0043] The scientific principle underlying the deoxidation (or purification) process of metal, alloy and intermetallic powders using calcium vapour in conjunction with a carrier substance is shown in FIG. 1(b) and is described in more detail below.

[0044] As shown in FIG. 1(b), the metal, alloy and intermetallic powder particles and metal oxide powder particles (a) are suspended in the liquid carrier (b) with Ca atoms dissolved in it. Ca vapour (c) is introduced to the top of the liquid carrier (b).

[0045] The liquid carrier is required to have some solubility of the Ca atoms, so some of the Ca atoms in the Ca vapour can enter the liquid carrier, and increase the content of dissolved Ca to the maximum level established by the thermodynamic equilibrium between the Ca vapour and the liquid carrier.

[0046] Once in the liquid carrier, the Ca atoms can interact with the metallic or intermetallic powder particles and the metal oxide particles suspended in the liquid carrier can react with the oxygen in the powder particles to form CaO and possibly other more complex metal oxide particles such as $\text{CaO} \cdot \text{Al}_2\text{O}_3$. The CaO and the $\text{CaO} \cdot \text{Al}_2\text{O}_3$ particles may be partially dissolved in the liquid carrier.

[0047] While some of the Ca atoms are consumed by the reactions, the content of the Ca dissolved in the liquid carrier is reduced to a below that established by the thermodynamic equilibrium between the Ca vapour and the liquid carrier.

[0048] The deficiency of Ca atoms in the liquid carrier will drive more Ca atoms in the Ca vapour to enter the liquid carrier. In this way, the supply of the Ca atoms to the reactions will be maintained and the reactions can continue until all the metal oxide powder particles and embedded inclusions are reduced and the content of dissolved oxygen in the metallic and intermetallic phases in the powder particles is reduced to a low level (e.g. <0.4 wt %).

[0049] The metal atoms produced by the reactions between the metal oxide phase and Ca atoms are combined with the metallic or intermetallic phases, or combine with themselves to form powder particles.

[0050] In preferred embodiments the reactive titanium rich powder particles together with CaO and $\text{CaO} \cdot \text{Al}_2\text{O}_3$ particles and carrier substance, CaCl_2 matrix are washed.

[0051] There are a number of available washing options and methods. These can include the use of room temperature or warm distilled water (40° C. to 50° C.) or diluted mild acidic solution (e.g. formic acid solution).

[0052] In preferred embodiments the washing leaches out the CaO and $\text{CaO} \cdot \text{Al}_2\text{O}_3$ particles and the solidified carrier substance such as CaCl_2 .

[0053] There are many advantages associated with this invention.

[0054] The advantage of generating Ca vapour for this reaction process is that low grade calcium can be purchased from suppliers such as Aldrich and therefore at low cost. However, when this low grade solid is converted into a vapour, it is of

very high purity. Also, the vapour can be added continuously to the titanium rich powder to provide a more efficient reaction and eliminates the steps of having to extract out any impurities in the solid.

[0055] The advantage of the use of a liquid carrier is that the titanium rich metallic or intermetallic powder particles are suspended in a liquid which can serve as a conductor for the deoxidant atoms to interact with the whole surface of each of the powder particles to be deoxidised, and thus allowing maximum effectiveness of the process. Specifically, the extraction of the dissolved oxygen and the oxygen associated with the metal oxide inclusions in the powder particles, and largely maintaining the original particle size and morphology. During holding the powder/carrier mixture at some temperatures, the powder particles may become more rounded, which is practically favourable for increased reaction surface.

[0056] This allows further reduction of the total oxygen content in a titanium rich powder to be below 0.4 weight %.

[0057] An advantage of this process is that it makes leaching of the reaction by-product, CaO and $\text{CaO} \cdot \text{Al}_2\text{O}_3$ particles much easier.

[0058] Also, a slight extension of the application of the process allows the use of a fluidised bed to replace the liquid carrier to achieve similar enhanced reaction kinetics between Ca atoms and titanium rich powder particles.

[0059] A further advantage of this process is that it can be used to purify simple binary Ti—Al intermetallic or alloy powders, but also can be used to purify more complex titanium based intermetallic or alloy powders as well as other metal based metal, alloy and intermetallic powders derived from a number of methods of manufacture.

BRIEF DESCRIPTION OF DRAWINGS

[0060] Further aspects of the present invention will become apparent from the following description which is given by way of example only and with reference to the accompanying drawings in which:

[0061] FIG. 1(a) Shows a diagrammatic representation of the reaction apparatus required for the generation and introduction of Ca vapour to the metallic or intermetallic powder/liquid carrier mixture;

[0062] FIG. 1(b) Shows a diagrammatic representation of the underlying principle of the method disclosed herein;

[0063] FIG. 2 Shows a diagrammatic representation of X-ray diffractometry pattern of the TiAl(O) powder to be deoxidised by the method disclosed herein;

[0064] FIG. 3 Shows a diagrammatic representation of scanning electron microscopy images (a) with magnified view (b) showing the cross sectional size, morphology and microstructure of particles in a TiAl(O) powder to be deoxidised by the method (the dark particles (A) embedded in the TiAl(O) matrix (B) in image (b) are Al_2O_3 inclusions);

[0065] FIG. 4 Shows a diagrammatic representation of an X-ray diffractometry pattern of a TiAl powder after being deoxidised;

[0066] FIG. 5 Shows a diagrammatic representation of scanning electron microscopy images (a) with magnified view (b) showing the cross sectional size, morphology and microstructure of the particles in a TiAl powder after being deoxidised (The dark regions inside the particles are pores);

[0067] FIG. 6 Shows a diagrammatic representation of an X-ray diffractometry pattern of a Ti—Al—V alloy powder to be deoxidised by the method disclosed herein;

[0068] FIG. 7 Shows a diagrammatic representation of scanning electron microscopy images (a) with magnified view (b) showing the cross sectional size, morphology and microstructure of particles in a Ti—Al—V powder to be deoxidised by the method (the dark particles (A) embedded in the Ti(Al,V,O)/Ti₃Al(O) matrix (B) in image (b) are Al₂O₃ inclusions);

[0069] FIG. 8 Shows a diagrammatic representation of an X-ray diffractometry pattern of a Ti—Al—V alloy powder to be deoxidised by the method disclosed herein;

[0070] FIG. 9 Shows a diagrammatic representation of Scanning electron microscopy images (a) with magnified view (b) showing the cross sectional size, morphology and microstructure of the particles in a Ti—Al—V alloy powder after being deoxidised;

[0071] FIG. 10 Shows a diagrammatic representation of an energy dispersive spectrum from Area 1 shown in FIG. 9 and corresponding contents of the elements determined from a semi-quantitative analysis based on the spectrum, and

[0072] FIG. 11 Shows a flow diagram of the specific steps required to carry out this invention.

BEST MODES FOR CARRYING OUT THE INVENTION

[0073] The steps (S1, S2, S3, S4, and S5) as shown in flow diagram FIG. 11 and detailed below utilise as a starting material, a titanium rich powder such as TiAl(O)/Al₂O₃ powder, Ti(Al,O)/Al₂O₃ powder, Ti(Al,V,O)/Al₂O₃ powder, Ti_xAl(O) powder, Ti(Al) powder or Ti(Al,V,O) formed by methods of manufacture known in the prior art base and that require further reduction of the oxygen content below 0.4 weight percent. Here, Al, O, and V in the brackets represent Al, O and V dissolved in the lattice of Ti or Ti_xAl phases.

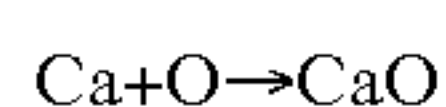
Step 1 Mixing the Titanium Rich Powder with the Carrier Powder

[0074] A titanium rich powder such as TiAl(O)/Al₂O₃ powder is mixed with a carrier powder such as hydrated calcium chloride (CaCl₂·2H₂O) powder. The volume ratio of the two powders should be such that the volume fraction of the titanium rich powder particles is preferably not higher than 60% of the total volume of the titanium rich powder particles and carrier powder particles.

Step 2 Introduction of Reactants

[0075] The mixture of the titanium rich intermetallic or metallic powder and the carrier powder is placed in a one chamber of a reaction container such as that, shown schematically in labelled FIG. 1(a).

[0076] A sufficient amount of calcium granules, pieces or powder is placed in the second chamber of the reaction container. The amount of calcium granules can be determined based on the total oxygen content of the powder, and the following chemical reaction equation:



[0077] Preferably the amount of calcium 10-20% in excess of the amount needed by the reaction.

Step 3 Generating Calcium Vapour and Introduction of the Vapour

[0078] The loaded reaction container with loose lid and an opening on the lid is placed in a stainless steel or heat resistant alloy retort which is then sealed. The retort is evacuated to a

pressure of less than 1 kPa, and then back filled with argon. This evacuation and back filling argon cycle is repeated for several times to reduce the content of air in the atmosphere, and then the retort is evacuated and maintained at a pressure of less than 1 kPa. While being evacuated, the retort is then heated to a temperature in the range of 250-350° C. and held at this temperature for 5 minutes to 2 hours, to allow the carrier substance to dehydrate and the water vapour and other volatile substances on the surface of the carrier to be removed. Then the retort is back filled with argon to a pressure of 40-100 kPa, heated to a desirable temperature in the range of 700-1400° C. and kept at the temperature for a desirable length of time in the range of 1 minute-100 hours. This will allow (a) calcium vapour to be generated from the evaporation of the calcium solid or liquid kept in one of the two chambers; (b) the carrier substance such as CaCl₂ to melt; (c) calcium vapour flow to the other chamber where the titanium rich powder/carrier liquid is kept; and (d) part of the calcium vapour to be dissolved in the carrier liquid.

Step 4 Reaction Between the Ca or CaH₂ Vapour and Inter-metallic Powder

[0079] While the retort is kept at the temperature specified in Step 3, the calcium atoms dissolved in the carrier liquid react with the titanium rich powder metallic and intermetallic powder particles and metal oxide particles suspended in the liquid carrier to extract the oxygen in them, forming CaO or other more complex oxide phase such as CaO·Al₂O₃ in the form of particles. The CaO and other metal oxide phases formed may be partially dissolved in the liquid carrier. These reactions can completely reduce the metal oxide powder particles and the metal oxide inclusions embedded in the titanium rich powder particles, and reduce the content of the oxygen dissolved in the metallic and/or intermetallic phases to below 0.4 twt %. The metal atoms such as Al atoms produced from the reactions between calcium and metal oxide such as Al₂O₃ will be alloyed with the titanium rich metallic and intermetallic powder particles.

Step 5 Washing the Powder

[0080] After cooling to room temperature or another sufficiently low temperature, the mixture of the treated titanium rich powder, the solidified carrier substance, the CaO particles and other metal oxide particles is crushed into granules, and washed using room temperature or warm distilled water (40° C. to 50° C.) or diluted mild acidic solution (e.g. hydrochloride or formic acidic solution) to leach out the carrier substance and the CaO and other metal oxide particles.

Example 1

[0081] 1 gram of TiAl(O) powder with a composition of approximately 69.6 wt % Ti-26.7 wt % Al-3.7 wt % O and containing Al₂O₃ inclusions was homogeneously mixed with 1.8 grams of CaCl₂·2H₂O powder. The X-ray diffractometry (XRD) pattern as shown in FIG. 2 shows that the powder mainly consists of TiAl(O), Ti₃Al(O) and Al₂O₃ phases. Here, O in the brackets represents the dissolved oxygen. FIG. 3 show the scanning electron microscopy (SEM) images of the cross sections of the powder particles. As shown in FIG. 3, Al₂O₃ inclusions are embedded in the TiAl based powder particles. The mixture of the TiAl(O) powder and the CaCl₂·2H₂O powder was placed in the top chamber of the reaction container as shown schematically in FIG. 1(a). 1 gram of calcium granules with a purity of 99% were placed in the bottom chamber of the reaction container. The container was

covered with a lid which has a small opening. The reaction container was placed in a stainless steel retort.

[0082] The retort was then sealed and placed in a tube furnace as shown schematically in FIG. 1(a). After this, the retort was evacuated and back filled with argon, and the cycle was repeated for 3 times. Then the retort is evacuated to a pressure of 1 kPa. While being evacuated, the retort is heated to 250° C. at a rate of 20° C./minute and held at the temperature for 10 minutes. Then the retort is back filled with argon to increase the pressure to 50 kPa, and heated to 1100° C. at a rate of 10° C./minute. The retort is then held at this temperature for 4 hours. At the end of the holding period, the furnace was switched off to allow the retort to be cooled to room temperature. The solidified CaCl_2 containing treated TiAl powder particles and the reaction products was taken out of the top chamber, crushed into small pieces and then washed using 60° C. warm water to leach out CaCl_2 , CaO and other oxide phases formed from the reactions. The XRD pattern of TiAl powder, as shown in FIG. 4, and the SEM images as shown in FIG. 5, show that the treated powder is free of Al_2O_3 . Chemical analysis of a sample from the treated powder showed that the oxygen content of the powder is 0.37 wt %.

Example 2

[0083] 1 gram of Ti—Al—V alloy powder with a composition of approximately 80 wt % Ti-10 wt % Al-5 wt % V-5 wt % O and containing Al_2O_3 inclusions was homogeneously mixed with 1.8 grams of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ powder. The XRD pattern as shown in FIG. 6 shows that the powder mainly consists of $\text{Ti}(\text{Al}, \text{V}, \text{O})$, $\text{Ti}_3\text{Al}(\text{O})$ and Al_2O_3 phases. Here, Al, V and O in the brackets represent Al, V and O dissolved in the titanium rich metallic and intermetallic phased. FIG. 7 shows the SEM images of the cross sections of the powder particles. As shown in FIG. 7, Al_2O_3 inclusions are embedded in the Ti—Al—V based powder particles. The mixture of the Ti—Al—V powder and the $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ powder was placed in the top chamber of the reaction container as shown schematically in FIG. 1(a). 1 gram of calcium granules with a purity of 99% were placed in the bottom chamber of the reaction container. The container was covered with a lid which has a small opening. The reaction container was placed in a stainless steel retort.

[0084] The retort was then sealed and placed in a tube furnace as shown schematically in FIG. 1(a). After this, the retort was evacuated and back filled with argon, and the cycle was repeated for 3 times. Then the retort is evacuated to a pressure of 1 kPa.

[0085] While being evacuated, the retort is heated to 250° C. at a rate of 20° C./minute and held at the temperature for 10 minutes. Then the retort is back filled with argon to increase the pressure to 50 kPa, and heated to 1100° C. at a rate of 10° C./minute. The retort is then held at this temperature for 4 hours. At the end of the holding period, the furnace was switched off to allow the retort to be cooled to room temperature. The solidified CaCl_2 together with the treated Ti—Al—V powder particles and the reaction product particles suspended in CaCl_2 was taken out of the top chamber, crushed into small pieces and then washed using 60° C. warm water to leach out CaCl_2 , CaO and other oxide phases formed from the reactions. The XRD pattern of TiAl powder, as shown in FIG. 8, and the SEM images as shown in FIG. 9, show that the treated powder is free of Al_2O_3 . Semi quantitative chemical analysis using the energy dispersive X-ray spectra generated from the cross sections of the powder par-

ticles, as shown in FIG. 10, shows that the average oxygen content of the Ti—Al—V powder particles is approximately 0.28 wt %.

[0086] Aspects of the present invention have been described by way of example only and it should be appreciated that modifications and additions may be made thereto without departing from the scope of the appended claims.

1. A method of purifying a metal comprising:
 - (a) placing the metal in powder form into a reaction apparatus;
 - (b) introducing to the metal powder a suitable carrier substance which can suspend the powder particles; and
 - (c) introducing a deoxidant calcium vapor into the reaction apparatus to create a reaction between the metallic powder and calcium vapor thereby removing inclusions in the metal.
2. A method as claimed in claim 1, wherein said calcium vapor is introduced through introducing calcium hydride vapor.
3. A method as claimed in claim 1, wherein particles of the metal in powder form are less than 100 micrometres.
4. A method as claimed in claim 1, wherein the inclusions are oxygen containing compounds.
5. A method as claimed in claim 4, wherein the inclusions are Al_2O_3 .
6. A method as claimed in claim 1, wherein the metal is a Ti_xAl compound.
7. A method as claimed in claim 1, wherein the metal powder is homogeneously mixed with the carrier substance.
8. A method as claimed in claim 7, wherein the carrier substance is calcium chloride (CaCl_2).
9. A method as claimed in claim 7, wherein the carrier substance is hydrated calcium chloride ($\text{CaCl}_2 \cdot \text{H}_2\text{O}$).
10. A method as claimed in claim 1, wherein calcium is a metal deoxidant in the form of a solid.
11. A method as claimed in claim 10, wherein the calcium solid is granules.
12. A method as claimed in claim 11, wherein the calcium granules are kept in separate chambers of the apparatus and heated to a temperature in the range of 700-1400° C.
13. A method as claimed in claim 12, wherein the calcium granules are heated at 700-1400° C. for a time period of 1 minute to 100 hours to allow generation of calcium vapor.
14. A method as claimed in claim 12, wherein the calcium vapor generated during heating is introduced to another apparatus chamber where the mixture of the metal and the carrier substance is kept and heated to the same temperature range of 700-1400° C.
15. A method of purifying a metal as claimed in claim 1, wherein the temperature allows the carrier substance to melt and a substantive fraction of the metal deoxidant to turn into vapor.
16. A method of purifying a metal as claimed in claim 6, wherein interactions between calcium atoms and the Ti_xAl powder particles are dispersed in the liquid carrier substance through a liquid medium.
17. (canceled)
18. A method as claimed in claim 2, wherein the inclusions are oxygen containing compounds.
19. A method as claimed in claim 3, wherein the inclusions are oxygen containing compounds.
20. A method as claimed in claim 2, wherein the metal is a Ti_xAl compound.
21. A method as claimed in claim 3, wherein the metal is a Ti_xAl compound.