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(71) **Demandeur/Applicant:**
INDUSTRIAL RESEARCH LIMITED, NZ

(72) **Inventeur/Inventor:**
OWERS, WILLIAM, NZ

(74) **Agent:** OYEN WIGGS GREEN & MUTALA

(54) **Titre** : PROCEDE DE PRODUCTION DE MATERIAUX DURCIS DE CARBURE DE TITANE, DE NITRURE DE TITANE, OU DE CARBURE DE TUNGSTENE

(54) **Title:** PROCESS FOR PRODUCING TITANIUM CARBIDE, TITANIUM NITRIDE, OR TUNGSTEN CARBIDE HARDENED MATERIALS

(57) **Abrégé/Abstract:**

A process for producing a composite material of titanium carbide, titanium nitride, or tungsten carbide and metal or a metal alloy such as iron or steel or an alloy comprises blending together precursor materials including a titanium or tungsten source, a source of carbon, and a metal or a metal alloy and heating the precursor materials in a vacuum or an inert atmosphere or a nitrogen containing atmosphere to a temperature effective to form TiC, TiN or WC, to thereby form a material comprising particles of TiC, TiN or WC substantially encapsulated in a matrix of metal or metal alloy.

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(71) **Applicant** *(for all designated States except US)*: **INDUSTRIAL RESEARCH LIMITED** [NZ/NZ]; Gracefield Road, Lower Hutt (NZ).

(72) **Inventor; and**
(75) **Inventor/Applicant** *(for US only)*: **OWERS, William** [NZ/NZ]; 14 Westhill Road, Point Howard, Wellington (NZ).

(74) **Agent: A J PARK**; 6th Floor, Huddart Parker Building, Post Office Square, P.O. Box 949, Wellington 6015 (NZ).

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PROCESS FOR PRODUCING TITANIUM CARBIDE, TITANIUM NITRIDE, OR TUNGSTEN CARBIDE HARDENED MATERIALS

FIELD OF INVENTION

The invention comprises a process for producing composite cermet materials comprising titanium carbide, titanium nitride or tungsten carbide and metal or metal alloy, suitable for the production of iron, steel or general metal or metal alloy components having high levels of hardness and wear resistance.

BACKGROUND

The term cermet is used to refer to materials that comprise both a ceramic and a metal or metal alloy, which are also known as metal matrix composites. Introducing a ceramic phase into a metal matrix provides characteristic features of each in the resultant product. The ceramic increases hardness and wear resistance but is often brittle, while the metal or metal alloy contributes toughness and ductility. However, "wetting" of the ceramic component by the metal to obtain cohesive bonding between the metal or metal alloy and the ceramic component is a major challenge in the preparation of such materials.

The commonly used commercial process for producing iron or steel components incorporating very hard ceramic substances such as titanium carbide for higher wear resistance requires sintering at high pressures and temperatures, which usually requires the use of an expensive hot isostatic press. Other routes have attempted *in-situ* synthesis of titanium carbide in steel by addition of ferrotitanium and/or metallic titanium to the molten steel. Hui et al in "Fabricating TiC Particulate Reinforced Fe-Cr-Ni Matrix Composites by a Liquid State In Situ Synthesis", Journal of Material Science Letters 19, 2000, 1281-1283 and in Jiang et al in "In-Situ Synthesis of a TiC-Fe Composite in Liquid Iron", Journal of Materials Science Letters 16 (1997) 1830-

1832 disclose processes for forming titanium carbide *in situ*, which involve addition of titanium filings to liquid iron for example.

New Zealand patent 229777 describes a process for the manufacture of cermets by the synthesis of titanium carbides and/or nitrides by carbothermal reduction of ilmenite.

SUMMARY OF INVENTION

It is the object of the present invention to provide an improved or at least alternative process for producing metal or metal alloy materials incorporating a hard ceramic substance.

In broad terms in a first aspect the invention comprises a process for producing a composite material of titanium carbide, titanium nitride, or tungsten carbide and a metal or metal alloy, comprising blending together precursor materials including a titanium or a tungsten source, a source of carbon, and a metal or metal alloy and heating the precursor materials in a vacuum or an inert atmosphere or a nitrogen containing atmosphere to a temperature effective to form TiC, TiN or WC, to thereby form a material comprising particles of TiC, TiN or WC substantially encapsulated in a matrix of iron.

Preferably the metal or metal alloy is iron or steel or an iron or steel alloy, but alternatively composites based on other metals such as copper or aluminium or magnesium and TiC, TiN or WC could be formed.

Preferably the titanium source includes titanium dioxide.

Preferably the iron source includes ilmenite, magnetite, or hematite, preferably of average particle size less than 10 microns.

In the process of the invention the precursor materials are heated to a temperature sufficient to form TiC, TiN or WC but at which the metal phase may soften but does not become molten (liquid) so that the TiC, TiN or WC is formed in situ without melting the metal phase. We have found that by the process of blending the precursor materials and then reacting at such a temperature cermets may be formed, in which the iron encapsulates the fine particles of titanium carbide, titanium nitride, or tungsten carbide which are formed, to hinder their surface oxidation on removal of the synthesised cermet bodies from the controlled atmosphere furnace. Further, the iron encapsulation assists wetting and dispersion of the ceramic component.

The process may include as a subsequent step adding the product produced to a melt of metal or a metal alloy to distribute the TiC, TiN or WC particles throughout the metal or metal alloy.

The product produced typically has a porous structure and the process may include as a subsequent step contacting the porous cermet with a molten metal or metal alloy so that the metal or metal alloy infuses into and fills voids within the porous cermet material.

The resulting material formed by the process of the invention may be useful as TiC, TiN or WC-rich intermediate billets or pellets for further processing into useful components, by hot metal forging for example. These TiC, TiN or WC-rich billets may also be added to molten iron, steel or iron alloy which is preferably under vacuum or an inert atmosphere to enable particulates of TiC, TiN or WC to be incorporated and distributed throughout the iron or steel or iron alloy. Alternatively, another process for producing end components may comprise synthesising the TiC-Fe, TiN-Fe or WC-Fe porous intermediate cermets as shapes or shaping same to the required component or product end shape, and then infilling or infusing the component as referred to, with metal or a metal alloy such as iron, steel or an iron alloy or alternatively aluminium or magnesium or their alloys, to form the final product or component.

BRIEF DESCRIPTION OF DRAWINGS

The accompanying figures are referred to in the further description. In the figures:

Figure 1 is the x-ray diffractogram of titanium carbide synthesised using the process of this invention and based on equation 5 showing the carbothermal reduction of titanium dioxide and ilmenite;

Figure 2 is a diffractogram of titanium nitride synthesised using the process of the invention and based on equation 7 showing the nitridation of titanium dioxide and ilmenite;

Figure 3 is a micrograph of a cross-section of a material produced as in the following example 5;

Figures 4 and 5 are micrographs of cross-sections of a material produced as in the following example 7;

Figure 6 is a micrograph of a material produced as in the following example 8; and

Figures 7 and 8 are micrographs of cross-sections of materials produced as in the following example 10.

DETAILED DESCRIPTION

In the process of the invention cermets rich in titanium carbide or tungsten carbide are produced by intimately blending together a titanium or tungsten source and a source of carbon and a metal or metal alloy, and heating the precursor materials in a vacuum or inert atmosphere to carbothermally reduce the titanium or tungsten source *in situ*, to thereby produce a porous cermet material rich in titanium carbide or tungsten carbide, bonded to and surrounded by a matrix of the metal or metal alloy. Alternatively cermets rich in titanium nitride may be produced by blending together a titanium source and a metal or a metal alloy and heating the precursor materials and carbon in a nitrogen containing atmosphere to produce a cermet material rich in titanium nitride. The reaction is carried out at a temperature which may soften the metal or metal alloy in the precursor material but does not cause the metal or metal alloy to become molten (liquid).

The precursor materials would typically be blended in a powdered or granular form, and by any suitable blending technique which will achieve intimate mixing of the precursor materials. For example the precursor materials may be mixed in a liquid medium and then dried (by which is meant removal of liquid by any means including oven drying or filtering for example) to form a substantially dry cake or body of the precursor materials, which is then heated to carry out the reaction. Alternatively the precursor materials may be mixed by being milled or ground together for example. For example titanium dioxide (or other titanium source) may be blended in an organic or aqueous medium with carbon preferably in powdered or particulate form, such as carbon lampblack, coal, charcoal or graphite or a material that decomposes to form carbon at an elevated temperature, with either clean fine particulate iron compounds such as ilmenite ($FeTiO_3$), magnetite (Fe_3O_4), hematite (Fe_2O_3) or metallic iron (Fe) produced by milling or a suitable alternative method. Preferably the carbon source is lampblack. Ideally ilmenite is used as a raw resource material because it contains titanium but any combination of iron compounds may be used. The molar quantities of titanium dioxide may vary widely but useful proportions are about three moles of titanium dioxide to about one of ilmenite.

After blending the precursor materials may then be shaped by being pressed or extruded for example, or by any other shaping operation(s) or form intermediate billets or pellets or to the final shape of a component or product. The precursor materials are then heated and reacted in a controlled temperature furnace under either vacuum or an inert atmosphere such as argon gas to synthesise titanium carbide. Alternatively heating under a nitrogen atmosphere will enable the synthesis of titanium nitride. The heating schedule comprises heating the shaped blended material in a non oxidising atmosphere to a temperature of at least substantially 1000°C and most preferably 1180 - 1450°C. Typical heating schedules for either the synthesis of titanium carbide or titanium nitride are 1180°C for 7 hours followed by 1250°C for 2 hours.

Equations 1 to 5 show carbothermal reactions of titanium dioxide with iron, ilmenite and hematite to synthesise titanium carbide. Equations 6 and 7 show the reaction for the synthesis of titanium nitride from ilmenite with and without the addition of titanium dioxide as a raw material.

$$Fe + TiO_2 + 3C = TiC + Fe + 2CO \qquad 1$$

$$Fe_2O_3 + 6TiO_2 + 21C = 6TiC + 2Fe + 15CO \qquad 2$$

$$FeTiO_3 + 4C = TiC + Fe + 3CO \qquad 3$$

$$FeTiO_3 + 2TiO_2 + 10C = 3TiC + Fe + 7CO \qquad 4$$

$$FeTiO_3 + 3TiO_2 + 13C = 4TiC + Fe + 9CO \qquad 5$$

$$FeTiO_3 + 3C + \tfrac{1}{2}N_2 = TiN + Fe + 3CO \qquad 6$$

$$FeTiO_3 + 3TiO_2 + 9C + 2N_2 = 4TiN + Fe + 9CO \qquad 7$$

Since the off gas from the synthesis reaction may contain carbon dioxide as well as carbon monoxide it may be necessary to deviate from the stoichiometric molar weights shown in the above equations to obtain phase pure resultant products.

Figure 1 is an x-ray diffractogram of the products of reaction equation 5. Figure 1 shows phase pure x-ray patterns characteristic of TiC and Fe.

Figure 2 is an x-ray diffractogram of the products of reaction equation 7. Figure 2 shows phase pure x-ray patterns characteristic of TiN and Fe.

The resulting carbothermally reduced porous cermet materials synthesised by the process of the invention are generally strong and readily handled without damage. Typically the synthesised material exhibits about 35-50% apparent porosity and there is a tendency for the outer boundary of the material to exhibit an enrichment of metallic iron. Whilst the material is porous the encapsulation of the titanium carbide, titanium nitride, or tungsten carbide, by the metallic iron matrix provides a protective barrier for these phases against surface oxidation. The exclusion of air by the metallic iron matrix hinders the oxidation at the surface of the TiC, TiN or WC particulates.

Titanium carbide, titanium nitride, or tungsten carbide in the enriched cermet intermediate material may be incorporated into metal or metal alloys such as iron or steel or iron or steel alloys by many methods including incorporating for example pellets of the material into molten metal and allowing the titanium carbide, titanium nitride, or tungsten carbide phase to disperse throughout the molten material. The encapsulation of the ceramic particulates of TiC, TiN and WC in the cermet intermediate by metallic iron prevents oxidation at the surface and enables ready wetting and cohesive bonding by the metal or metal alloy. A vacuum induction furnace with provision for supply of a protective atmosphere such as argon is the preferred production method and such heating is often accompanied by significant stirring and therefore rapid mixing of the solid TiC, TiN or WC particulates could be expected.

An alternative and useful method to prepare titanium carbide, titanium nitride, or tungsten carbide enriched billets is by allowing molten metal such as iron or steel or iron or steel alloys, or aluminium or magnesium or their alloys, to infill the porous titanium carbide, titanium nitride or tungsten carbide enriched material by capillary attraction.

The process of the invention is further illustrated by the following examples:

Example 1

60 grams of iron powder, 108 grams of carbon black and 239.7 grams of titanium dioxide were blended together in ethanol, excess ethanol was removed by filtration, and the resulting filter cake was extruded, dried and then heated under vacuum at 1180°C for 12 hours followed by 1250°C for 2 hours. X-ray diffraction patterns of the reacted product showed phase pure titanium carbide and iron.

Example 2

63.9 grams of hematite (Fe_2O_3), 100.9 grams of carbon black and 191.7 grams of titanium dioxide were blended together in ethanol, excess ethanol was removed by filtration, and the resulting filter cake was extruded, dried and then heated under vacuum at 1180°C for 12 hours followed by 1250°C for 2 hours. X-ray diffraction patterns of the reacted product showed phase pure titanium carbide and iron.

Example 3

152 grams of ilmenite, 117.8 grams of carbon black and 160 grams of titanium dioxide were blended together in ethanol, excess ethanol was removed by filtration, and the resulting filter cake was extruded, dried and then heated under vacuum at 1180°C for 14 hours followed by 1250°C for 2 hours. X-ray diffraction patterns of the reacted product showed phase pure titanium carbide and iron.

Example 4

152 grams of ilmenite, 153.8 grams of carbon black and 240 grams of titanium dioxide were blended together in ethanol, excess ethanol was removed by filtration, and the resulting filter cake was extruded, dried and then heated under vacuum at 1180°C for 14 hours followed by 1250°C for 2 hours. X-ray diffraction patterns of the reacted product showed phase pure titanium carbide and iron.

Example 5

4.66 grams of TiC enriched material produced as in example 4 was placed in an alumina crucible with 9.91 grams of NiHard alloy (BSS 4844 Grade 2D) and these materials were heated under a protective argon atmosphere to 1450°C for two hours (without stirring). After cooling the material was found to have taken the form of the crucible and the cross section of the body showed that TiC had dispersed throughout the whole body. Figure 3 shows a micrograph of a cross section of the material. Initial hardness of the NiHard alloy used in the process was 450 HV_{20} and the hardness of the resultant material with the incorporation of TiC was 808 HV_{20}.

Example 6

4.57 grams of TiC enriched material produced as in example 4 was placed in an alumina crucible with 9.11 grams of NiHard alloy (BSS 4844 Grade 2D) and these materials were heated under a protective argon atmosphere to 1450°C for two hours (without stirring). After cooling the material was found to have partially taken the form of the crucible and the cross section of the body showed that the TiC had dispersed throughout the whole material. Initial hardness of the NiHard alloy used was 450 HV_{20} and the hardness of the resultant material with the incorporation of TiC was 850 HV_{20}.

Example 7

8.37 grams of TiC enriched material produced as in example 4 was placed in an alumina crucible with 50.23 grams of NiHard alloy (BSS 4844 Grade 2D) and these materials were heated under a protective argon atmosphere to 1500°C for two hours without stirring. After cooling the molten material was found to have taken the form of the crucible and the cross section of the body showed that the TiC had dispersed throughout 73% of the body of the material. The initial hardness of the NiHard alloy used was 450 HV_{20} and the portion of the final material containing TiC had a mean hardness measurement of 708 HV_{20}. Percentage area measurement of the TiC particulates on a cross section of this sample was 38.9 as determined using SEM back scatter imaging. Figures 4 and 5 are micrographs of the sample portion containing TiC.

Example 8

2.71 grams of TiC enriched material produced as in example 4 was placed in an alumina crucible with 2.71 grams of NiHard alloy (BSS 4844 Grade 2D) and these materials were heated under a protective argon atmosphere to 1450°C for two hours. After cooling the molten material was found to have infilled the extruded body to the complete length by capillary attraction. The extruded material retained its original form after heating to 1450°C and further processing into useful high TiC components is possible by hot forging. The initial hardness of the NiHard alloy used was 450 HV_{20} and the material infilled with TiC gave a mean hardness measurement of 1000 HV_{20}. Percentage area measurement of the TiC particulates on a cross section of this sample was 52.3 as determined using SEM back scatter imaging. Figure 6 shows a micrograph of this sample.

Example 9

151.7 grams of ilmenite, 120 grams of carbon black and 159.8 grams of titanium dioxide were blended together in ethanol, excess ethanol was removed by filtration, and the partially dried filter cake was extruded, dried and then heated under vacuum at 1180^{0}C for 7 hours followed by1250^{0}C for two hours. X-ray diffraction patterns of the reacted products showed phase pure titanium carbide and iron.

5.42 grams of resulting TiC enriched material was then placed in an alumina crucible together with 10.87 grams of NiHard alloy (BSS 4844 Grade 2D) and heated under vacuum to a soak temperature of 1400^{0}C for 3 hours. After cooling the heated material was found to have taken the form of the crucible and a cross section of the body showed that the TiC had dispersed throughout the whole body with a mean hardness of 719 HV_{20}. This material was then reheated under a protective argon atmosphere to a soak temperature of 1560^{0}C for 3 hours and when cooled this reheated material was found to have increased in hardness to a mean value of 898 HV_{20}.

Example 10

176.7 grams of TiC enriched material produced as in example 4 was placed in a fireclay crucible with 353.4 grams of NiHard alloy (BSS 4844 Grade 2D) and these materials were heated under vacuum to 1400^{0}C for a 3 hour soak period. After cooling, the molten material was found to have taken the form of the crucible and formed a non-porous body by infilling the previously porous TiC material with NiHard alloy.

This non porous TiC containing billet of 515 grams was placed together with 1905 grams of NiHard alloy (BSS 48444 Grade 2D) into a crucible of a 10 kg capacity induction furnace. A small quantity of slag (40wt%CaO/40wt%Al_2O_3/20wt %SiO_2)

was added. These materials, under a vacuum of 1.5 Torr, were electrically heated initially at 8kW power input which was increased stepwise to 17 kW over 21 minutes. After 31 minutes when the material was molten and at 1550^{0}C, the chamber was opened and the melt stirred. The melt was manually stirred for a further five occasions over the following 15 minutes and after the last stirring 46 minutes from commencement of the heating, the melt was poured into a sand mould. There were risers of 7, 10, and 13 mm diameter within the sand mould. Microscopic examination of these cross sections showed distribution of TiC throughout the metal matrix. Micrographs of two such examples are shown in figures 7 and 8.

Example 11

Samples of grey cast iron with and without various portions of TiC enriched material prepared as in example 9 (first part) were heated initially to 1440^{0}C under vacuum and then to 1500^{0}C under an argon atmosphere. One part by weight of enriched TiC was added to 20, 10 and 5 parts by weight of the cast iron. The cast iron specimen heated without the presence of TiC showed significant grain growth but such grain growth was inhibited on all three specimens with added TiC.

The mean pyramid diamond hardness values of specimens of the heated cast iron and those with increasing TiC content are shown below.

TiC : cast iron, (pbw)	HV_{10}
cast iron only	109
1:20	170
1:10	258
1:5	257

Example 12

TiC enriched material as in example 9 (first part) was placed in a graphite crucible together with aluminium alloy and heated under vacuum to a soak temperature of 900°C for 1 hour. After cooling the aluminium was found to have infilled the body to the complete length by capillary attraction producing a dense and light weight cermet. X-ray diffraction patterns of the reacted product showed titanium carbide, aluminium and iron aluminide phases The initial hardness of the aluminium alloy used was 35 HV_{20} and the infilled material containing 45 volume percent of TiC gave a mean hardness measurement of 110 HV_{20}.

The foregoing describes the invention including preferred forms thereof. Alterations and modifications as will be obvious to those skilled in the art are intended to be incorporated in the scope hereof as defined in the accompanying claims.

CLAIMS:

1. A process for producing a composite material of titanium carbide, titanium nitride, or tungsten carbide and metal or a metal alloy, comprising blending together precursor materials including a titanium or a tungsten source, a source of carbon, and a metal or a metal alloy and heating the precursor materials in a vacuum or an inert atmosphere or a nitrogen containing atmosphere to a temperature effective to form TiC, TiN or WC, to thereby form a material comprising particles of TiC, TiN or WC substantially encapsulated in a matrix of metal or metal alloy.

2. A process according to claim 1 wherein the metal or metal alloy is iron or steel or an iron or steel alloy.

3. A process according to either one of claims 1 and 2 for producing a composite material comprising WC including carrying out said heating a vacuum or in an inert atmosphere.

4. A process according to either one of claims 1 and 2 for producing a composite material comprising TiN including carrying out said heating in a nitrogen containing atmosphere.

5. A process according to either one of claims 2 and 4 wherein the titanium source includes titanium dioxide.

6. A process according to either one of claims 2 and 4 where the titanium and iron source includes ilmenite, magnetite, or hemamite.

7. A process according to any one of claims 1 to 6 wherein the carbon source includes carbon black.

8. A process according to any one of claims 1 to 7 including blending all of the precursor materials in powdered or granular form before said heating.

9. A process according to claim 8 including blending the precursor materials in a liquid medium and then drying the precursor materials.

10. A process according to claim 8 including blending the precursor materials by milling or grinding the precursor materials together.

11. A process according to any one of claims 1 to 10 including forming a shaped body of the precursor materials after blending but before heating the precursor materials.

12. A process according to claim 11 wherein said shaped body is a billet or pellet.

13. A process according to claim 11 wherein said shaped body is substantially in the final shape of a component or product.

14. A process according to any one of claims 1 to 13 comprising carrying out said heating to a temperature in the range 1180 to 1450°C.

15. A process according to any one of claims 1 to 14 including the subsequent step of adding the product produced to a melt of metal or a metal alloy to distribute the TiC, TiN or WC particles throughout the metal or metal alloy.

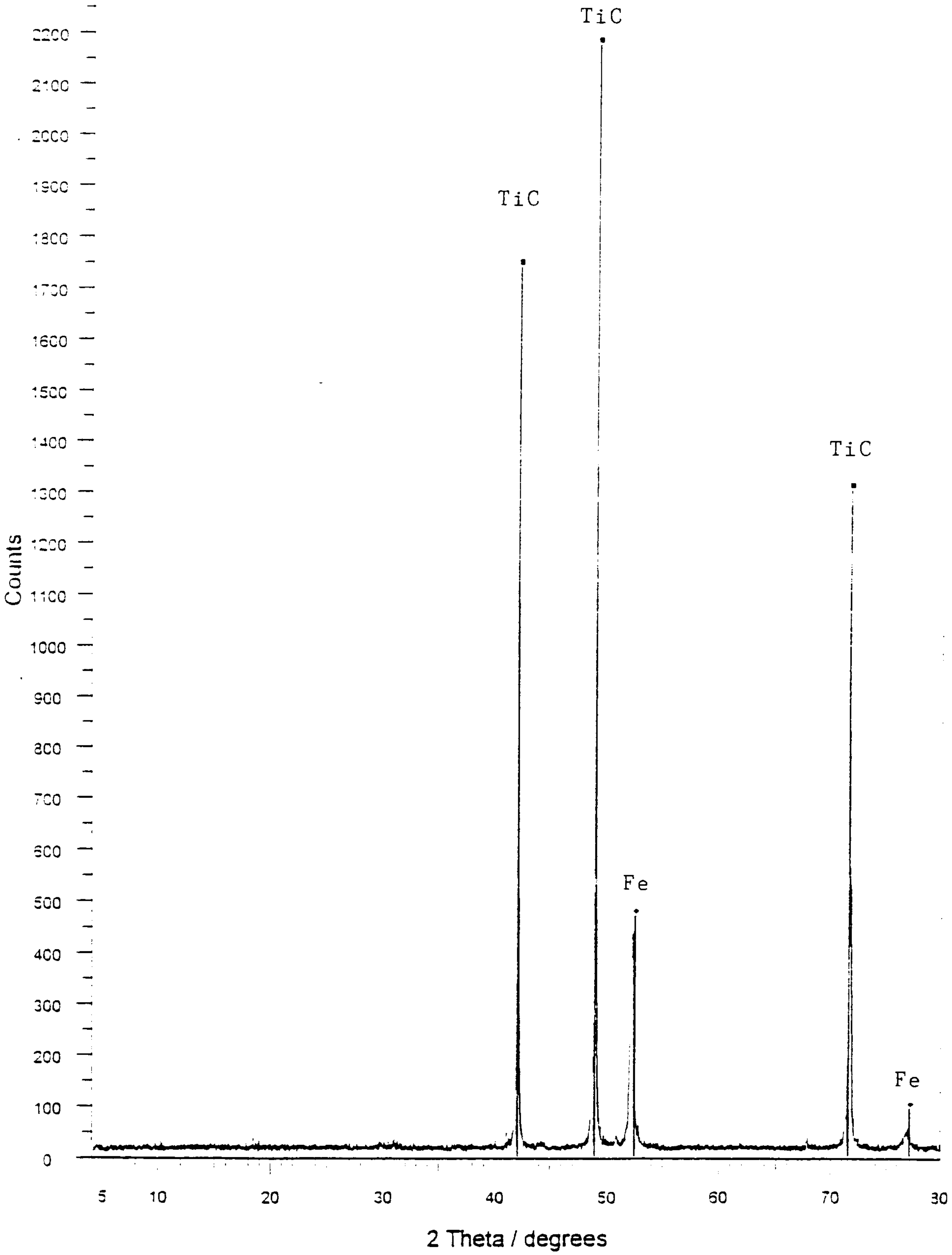
16. A process according to any one of claims 1 to 14 wherein said composite materials are porous and optionally pre-shaped and including the subsequent step of contacting the porous cermet with a molten metal or metal alloy so

that the metal or metal alloy infuses into and fills voids within the porous cermet material.

17. A process according to claim 16 wherein said metal which infuses into and fills voids within the porous cermet material includes iron or steel or an iron or steel alloy.

18. A process according to claim 16 wherein said metal which infuses into and fills voids within the porous cermet material includes aluminium or magnesium or an aluminium or magnesium alloy.

19. A composite material of titanium carbide, titanium nitride, or tungsten carbide and metal or a metal alloy, produced by a process as claimed in any one of the preceding claims.


TiC
TiC
TiC
Fe
Fe
Counts
2200
2100
2000
1900
1800
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
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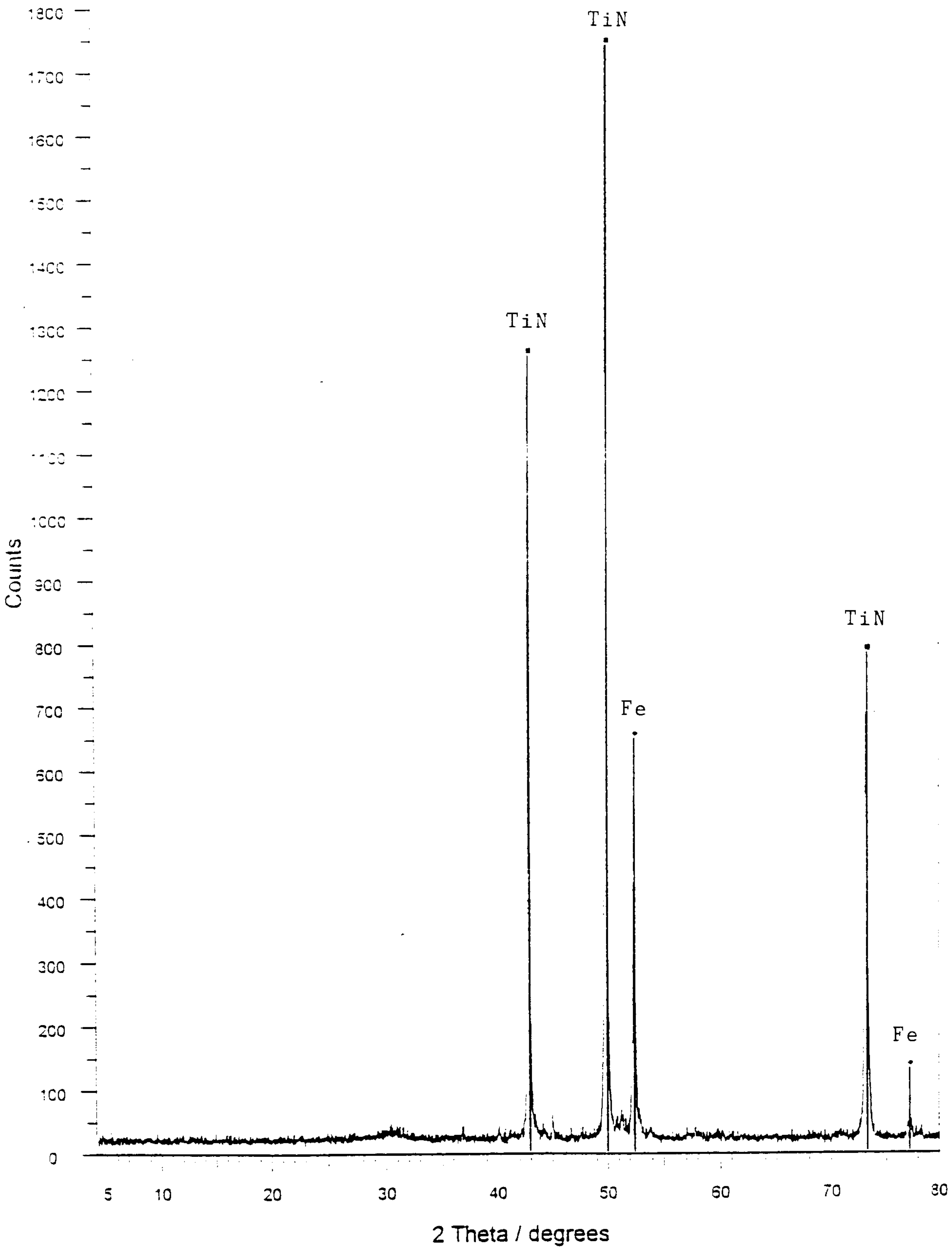
70
80
2 Theta / degrees

Figure 1


TiN
TiN
Fe
TiN
Fe
Counts
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1700
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1400
1300
1200
1100
1000
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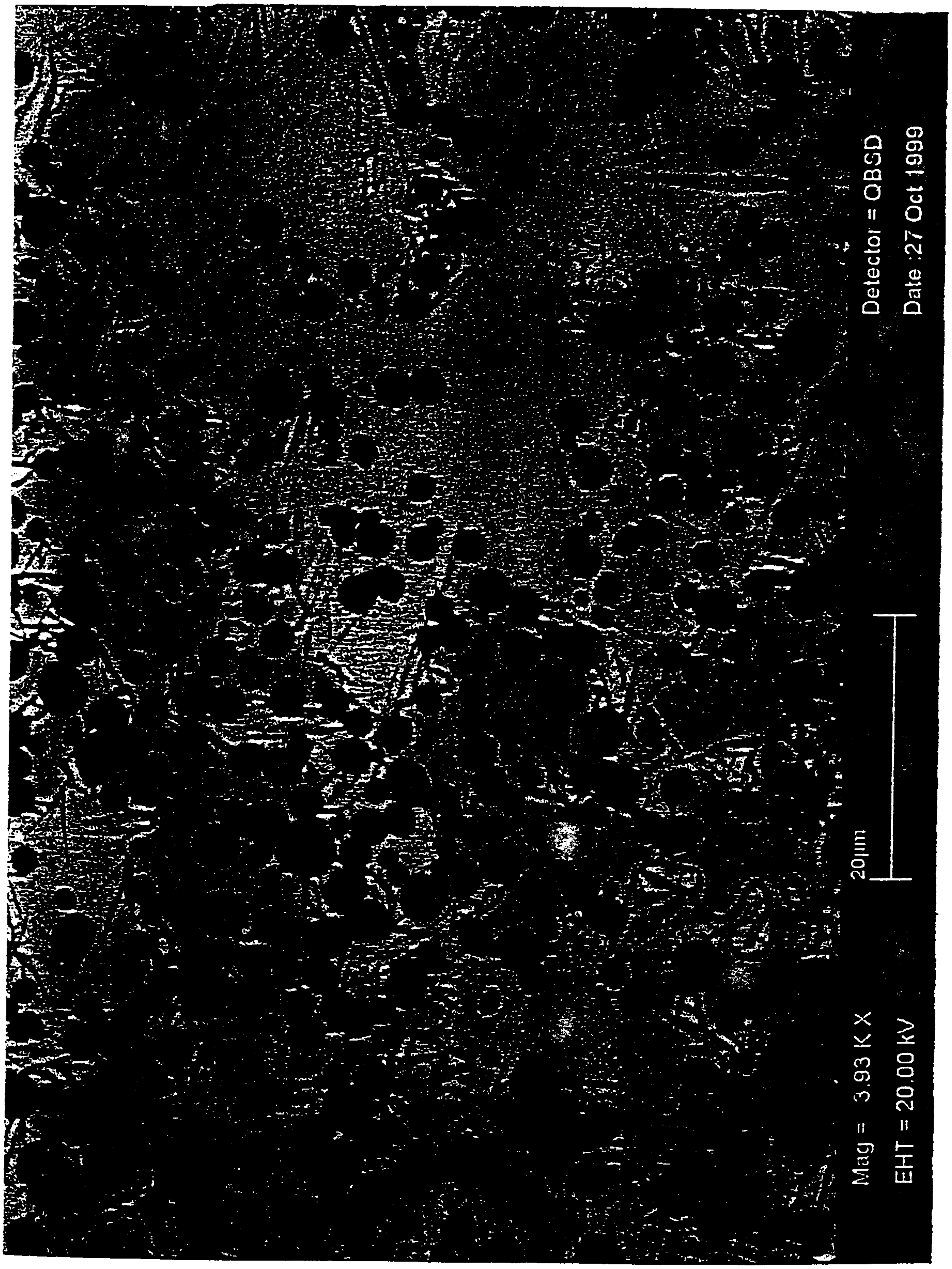
Figure 2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Date :27 Oct 1999
20µm
Mag = 3.93 K X
EHT = 20.00 kV
Ni4a

Figure 3

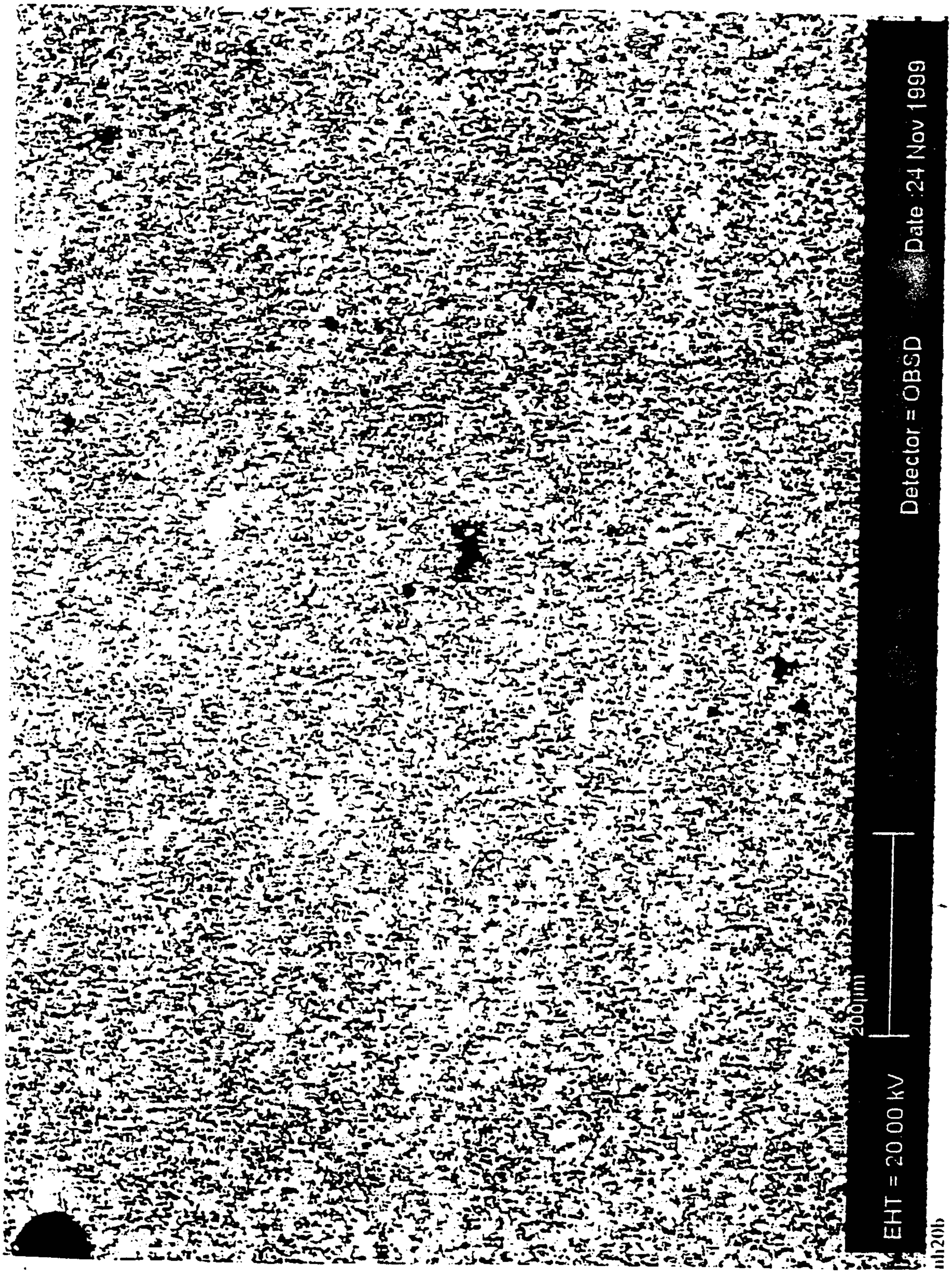


Figure 4

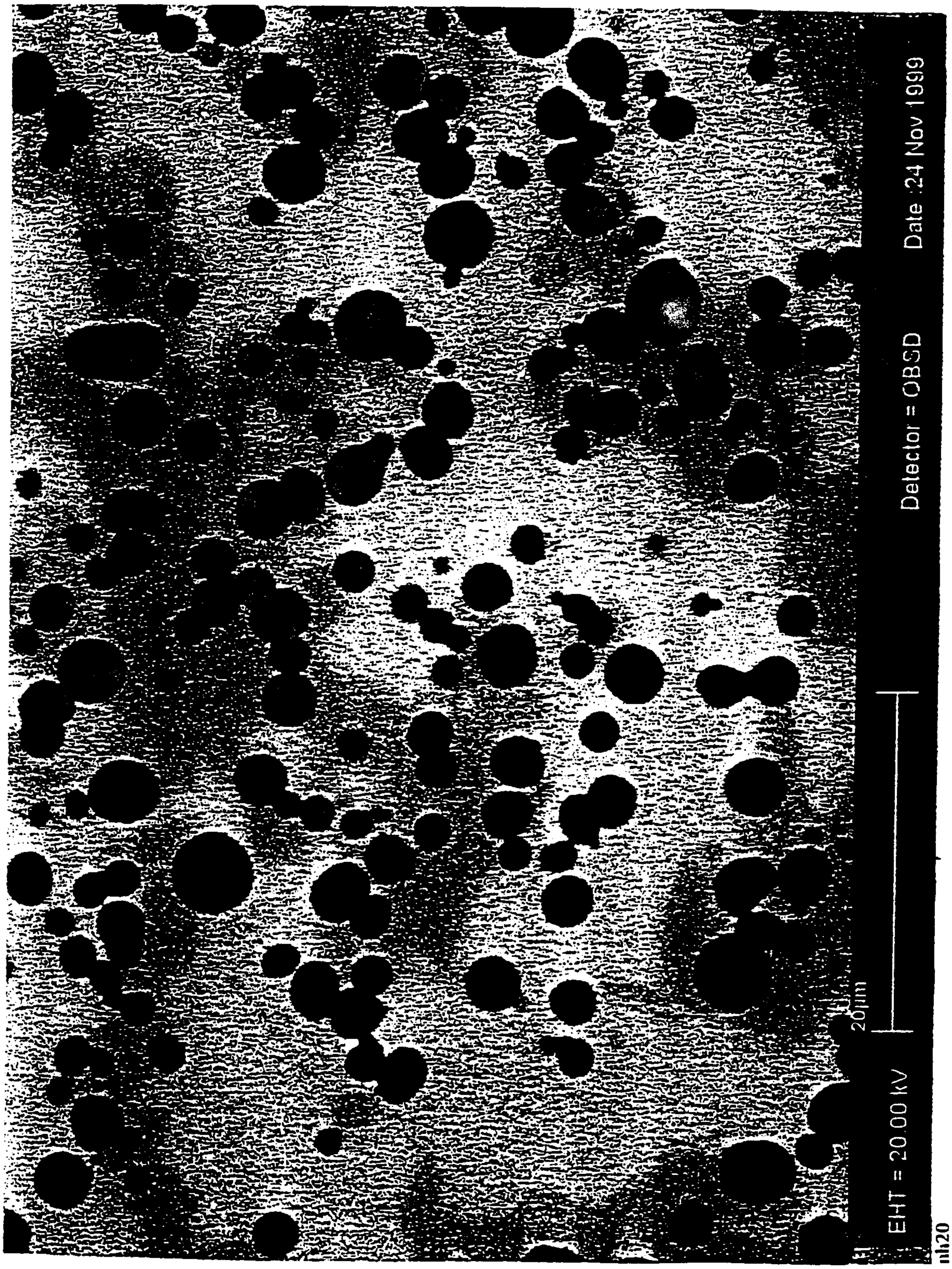


Figure 5

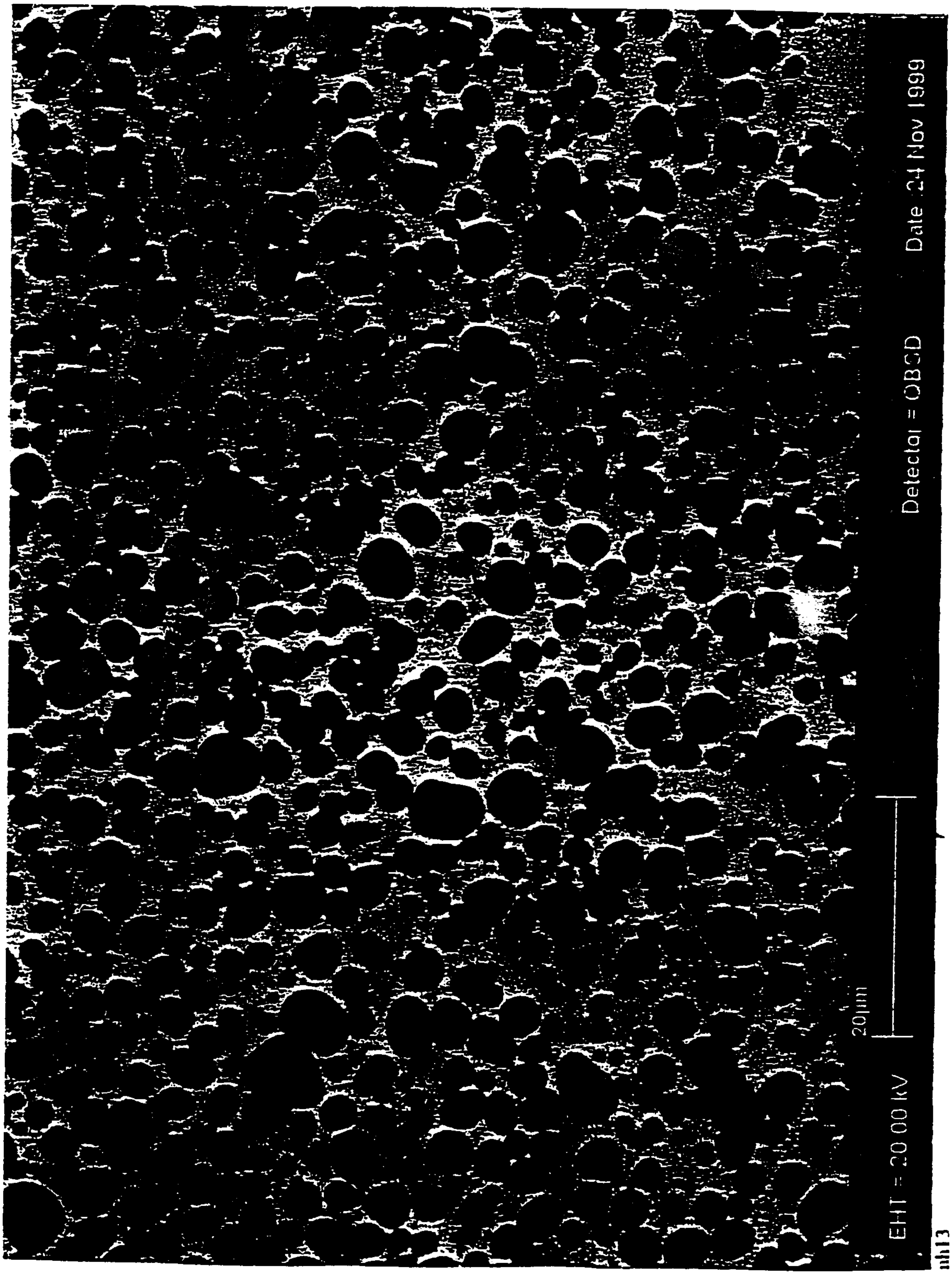
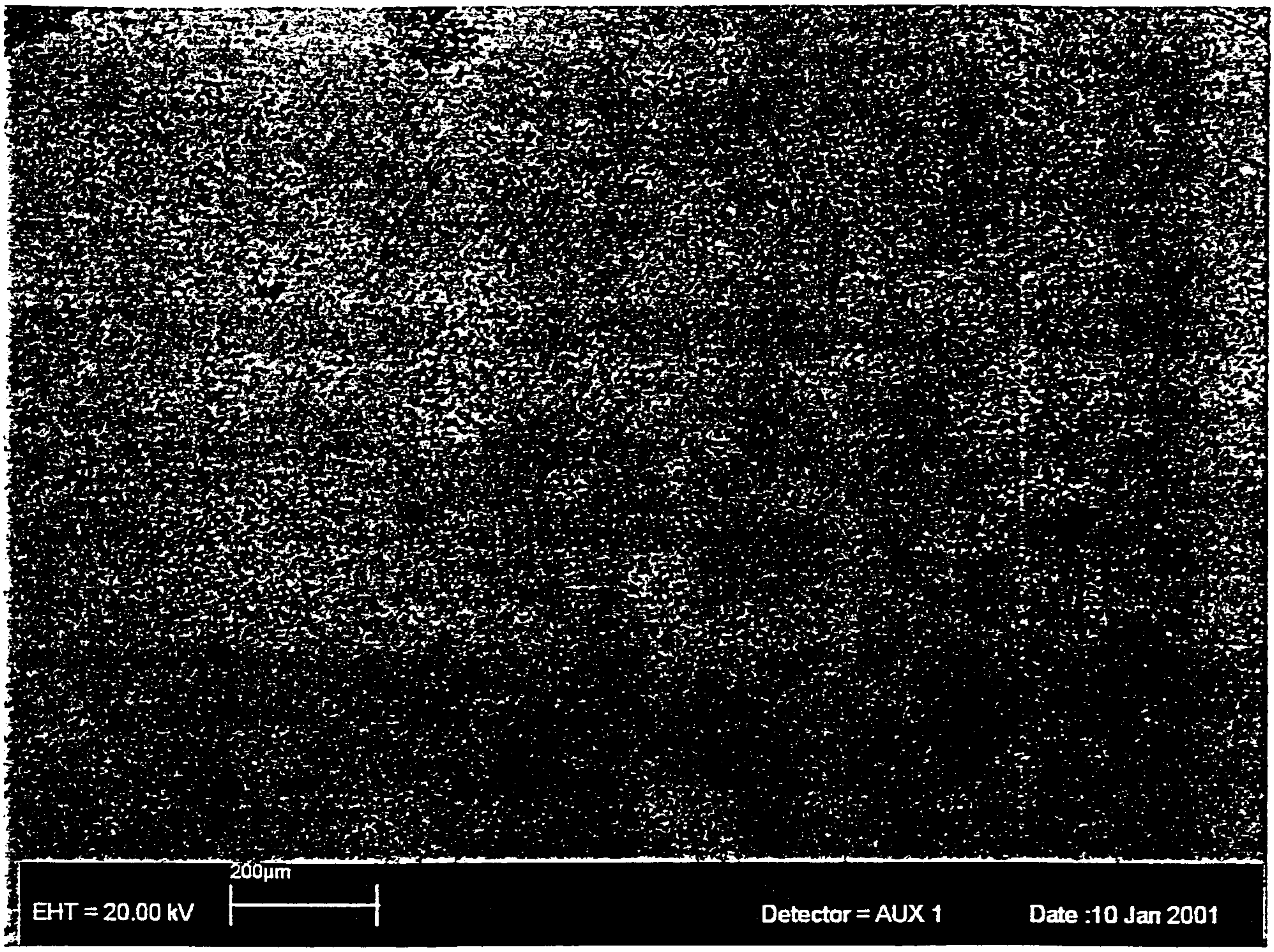


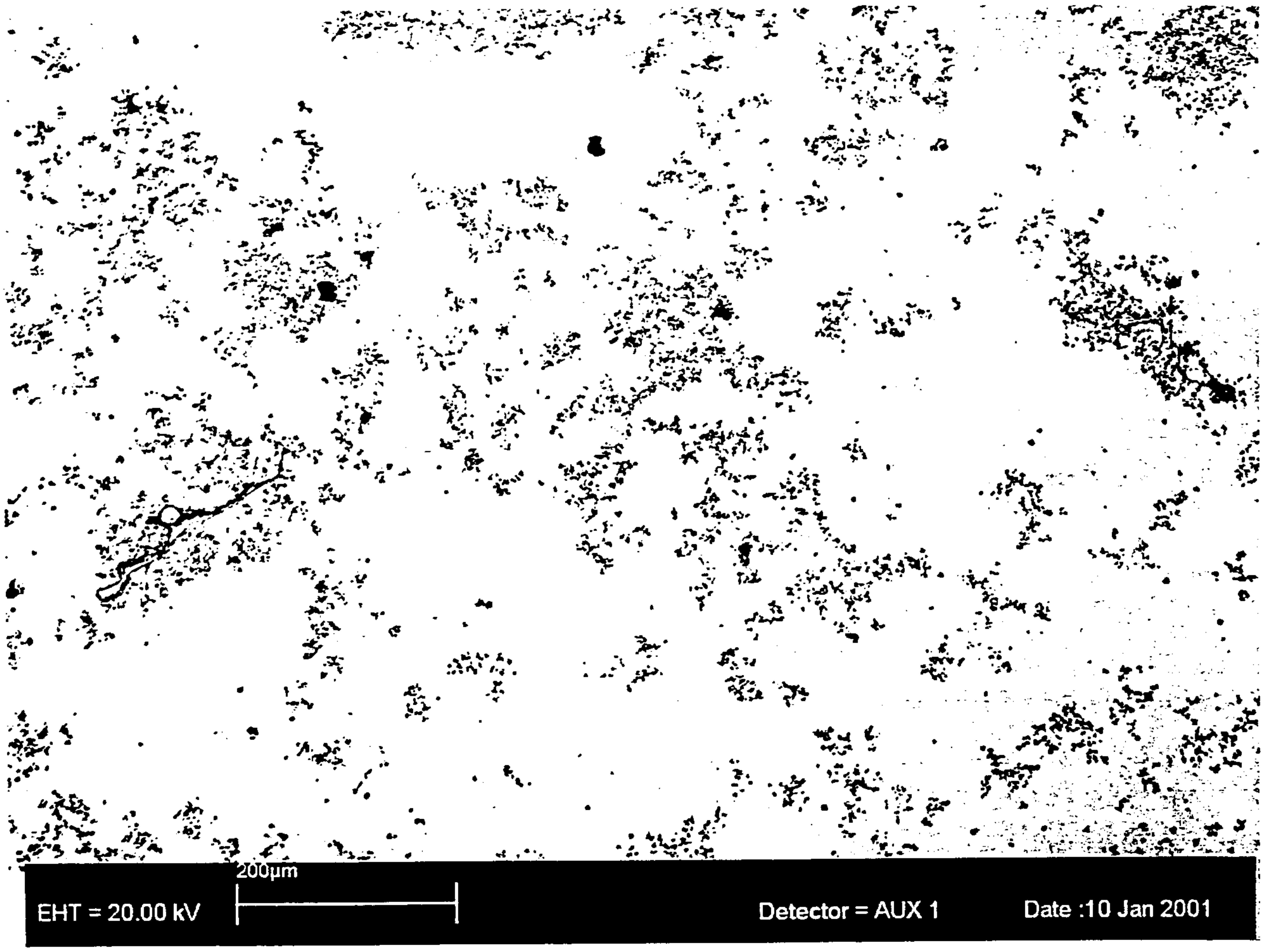
Figure 6



Area Measurements;

TiC	06.6%
Metal matrix	92.4%
Other	01.1%

Figure 7



Area Measurements:

TiC	03.5%
Metal matrix	96.1%
Other	00.4%

Figure 8