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(54) Title: FLAKED TANTALUM POWDER AND METHOD OF USING SAME
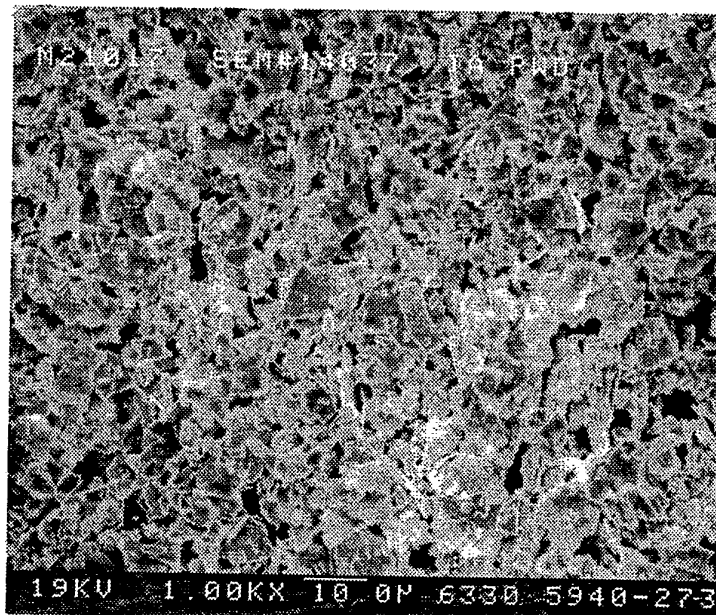
(57) Abstract

An improved flaked tantalum powder and process for making the flaked powder are disclosed. The powder is characterized by having a mean particle size in the range of about 2 to about 55 micrometers and a BET surface area in the range of about 0.5 to about 5.0 m²/g. Preferably, the mean particle size and BET surface area are selected so that the flakes have an aspect ratio (D/T) in the range of about 2 to about 50. Agglomerates of the flaked tantalum powder, provide improved flowability, green strength and pressing charactistics compared to conventional flaked tantalum powders. The improved flaked tantalum powder can be made by preparing a flaked tantalum and then reducing the flake size until such mean particle size and BET surface areas are achieved such that the flakes have an aspect ratio (D/T) in the range of about 2 to about 50.

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FLAKED TANTALUM POWDER AND METHOD OF USING SAME
CROSS-<u>REFERENCE TO RELATED APPLICATIONS</u>

This application is a continuation-in-part of Ser. No.
5   462,806, Jan. 10, 1990, which is a division of Ser. No.
209,746, Jun. 4, 1988, now U.S. Patent No. 4,940,490,
which is a continuation-in-part of Ser. No. 126,706, Nov.
30, 1987, now abandoned.

<u>FIELD OF INVENTION</u>

10   The invention relates to flaked tantalum powders often
used for electrical capacitors and methods for using such
powders to make low density pellets used for making
capacitors. More particularly, the invention relates to
flaked tantalum powders, having a specified aspect ratio,
15   which when agglomerated provide the required good
processing properties, e.g., flowability, high green
strength and pressability. These flaked tantalum powders
can also make capacitors having low leakage and high
breakdown voltages.

20

<u>BACKGROUND OF THE INVENTION</u>

Tantalum capacitors, made from tantalum powder, have been
a major contributor to the miniaturization of electronic
circuits and have made possible the application of such
25   circuits in extreme environments. Tantalum capacitors
typically are manufactured by compressing agglomerated
tantalum powder to form a pellet, sintering the pellet in
a furnace to form a porous tantalum body (electrode), and
then subjecting the porous body to anodization in a
30   suitable electrolyte to form a continuous dielectric
oxide film on the sintered body.

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Development of powders suitable for making tantalum
capacitors has resulted from efforts by both capacitor
producers and tantalum processors to delineate the
characteristics required for tantalum powder in order for

5   it to best serve in the production of quality capacitors.
Such characteristics include specific surface area,
purity, shrinkage, pressability, green strength, and
flowability.

10   First of all, the powder should provide an adequate
electrode surface area when formed into a porous body and
sintered. The μfV/g of tantalum capacitors is
proportional to the specific surface area of the sintered
porous body produced by sintering a tantalum powder

15   pellet; the greater the specific surface area after
sintering, the greater the μfV/g. The specific surface
area of tantalum powder is related to the maximum μfV/g
attainable in the sintered porous body.

20   Purity of the powder is an important consideration.
Metallic and non-metallic contamination tends to degrade
the dielectric oxide film in tantalum capacitors. While
high sintering temperatures serve to remove some volatile
contaminants high temperatures tend to shrink the porous

25   body reducing its net specific surface area and thus the
capacitance of the resulting capacitor. Minimizing the
loss of specific surface area under sintering conditions,
i.e., shrinkage, is necessary in order to produce high
μfV/g tantalum capacitors.

30
Flowability of the tantalum powder and green strength
(mechanical strength of pressed unsintered powder
pellets) are also important characteristics for the
capacitor producer in order to provide efficient

35   production. The flowability of the agglomerated tantalum
powder is essential to proper operation of automatic
pellet presses. Sufficient green strength permits

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handling and transport of a pressed product, e.g.,
pellet, without excessive breakage.

5   A 'pellet', as the term is used herein, is a porous mass
    or body comprised of tantalum particles. Green strength
    is a measure of a pellet's mechanical strength. The term
    'pressability' describes the ability of a tantalum powder
    to be pressed into a pellet. Tantalum powder that forms
    pellets that retain their shape and have sufficient green
10 strength to withstand ordinary processing/manufacturing
    conditions without significant breakage have good
    pressability.

    Currently, tantalum powders suitable for use in high
15 performance capacitors are produced by several methods.
    One powder production method involves chemical reduction,
    e.g., sodium reduction of potassium fluorotantalate,
    K_2TaF_7. In another method, powder is produced by
    hydriding a melted (typically arc melted or electron beam
20 melted) tantalum ingot, milling the hydrided chips, and
    dehydriding.

    As discussed above, the $\mu fV/g$ of a tantalum pellet is a
    function of the specific surface area of the sintered
25 powder. Greater net surface area can be achieved, of
    course, by increasing the quantity (grams) of powder per
    pellet; but, cost and size considerations have dictated
    that development be focused on means to increase the
    specific surface area of tantalum powder.
30
    One of the methods proposed for increasing the specific
    surface area of tantalum powder is flattening the powder
    particles into a flake shape.

35 Efforts to increase specific surface area by making
    thinner tantalum flakes have been hindered by concomitant
    loss of processing characteristics, for example, very

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thin tantalum flake would be expected to have poor flow
characteristics, poor pressability, low green strength
and low forming voltages. Thinner flakes are desirable
however, because they could make less expensive low
5   density pellets which could be used to make capacitors
having high breakdown voltages and low leakage losses.

OBJECTS AND SUMMARY OF THE INVENTION

It is an object of this invention to provide a method for
10 making a fractured flake tantalum powder having BET
surface area and mean particle size represented by a
range of aspect ratios (D/T) of about 2 to about 50.

It is another object of this invention to provide a
15 fractured flake tantalum powder with a BET surface area
greater than 0.7 m^2/g and a mean particle size in a range
selected to yield an aspect ratio (D/T) that has powder
properties suitable for use in economical high speed
processes for the manufacture of tantalum capacitors;
20 e.g., an aspect ratio in the range of about 2 to 50.

It is another object of this invention to provide an
agglomerate of flaked tantalum powder that has good
flowability and pressability characteristics.
25
It is another object of this invention to provide low
density flaked tantalum pellets having high green
strength.

30 It is another object of this invention to provide low
density flaked tantalum pellets having reduced
sensitivity to sintering temperatures, i.e., pellets that
can be sintered over a wide range of temperatures,
relative to the prior art, to form an electrode useful in
35 a tantalum capacitor.

5

It is another object of this invention to provide a
tantalum electrode having reduced sensitivity to forming
voltages, i.e., dielectric oxides can be formed on the
electrodes over a range of voltages.

5

It is another object of this invention to provide a
tantalum electrode having low leakage and high breakdown
voltages.

10   The present invention provides a method for making flaked
tantalum powder that is flowable and pressable when
agglomerated, comprising the steps of:
        preparing tantalum flake powder;
        reducing the flake size until the powder has a mean
15   particle size in the range of about 2 to about 55
micrometers and BET surface area in the range of about
0.5 to about 5.0 m^2/g and
            selecting said mean particle size and BET
surface area so that the flakes have an aspect ratio
20   (D/T) in the range of about 2 to about 50.

The present invention also provides a fractured flake
tantalum powder comprised of flakes having a mean
particle size in the range of 2 to 55 micrometers and a
25   BET surface area greater than about 0.7 m^2/g and an aspect
ratio between about 2 and about 50.

The present invention also provides an agglomerate of the
above-described flaked tantalum powder having improved
30   flowability and pressability characteristics. The
agglomerated flaked tantalum powder of this invention may
be prepared by any conventional method for preparing
agglomerates such as, for example, by heating the
tantalum flake, described in the preceding paragraphs, to
35   a temperature of about 1300 to 1600°C in an inert
atmosphere or under vacuum for a period of about 30 to 60
minutes and crushing the resulting product to a size in

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the range of about 40 mesh (0.015 inch screen opening) to
60 mesh (0.0098 inch screen opening).

The present invention also provides low density pellets
5   prepared from the flaked tantalum powder and/or
agglomerate described in the preceding paragraphs.

The present invention also provides a capacitor electrode
formed from the pellets described in the preceding
10 paragraphs. In general the capacitors are prepared by
sintering the pellets described above and anodizing the
sintered pellets.

BRIEF DESCRIPTION OF THE DRAWINGS

15

Other details, objects and advantages of the invention
and methods for making and using the same will become
apparent from the following detailed description and
accompanying Figures. A legend at the bottom of the
20 Scanning Electron Micrograph (SEM) Figures gives the
voltage, magnification e.g., 400X, and a reference scale
in micrometers.

Figure 1 is a SEM at a magnification 1000X, of a prior
25 art ingot-derived flaked tantalum powder having a Scott
Density of 13.4 g/in^3;

Figure 2 is a SEM at a magnification of 1000X, of an
ingot-derived flaked tantalum powder having a Scott
30 Density of 59.8 g/in^3 prepared in accordance with the
present invention;

Figure 3 is a SEM at a magnification of 1000X, of a prior
art flaked tantalum powder produced in accordance with
35 Example II, specimen H of U.S. Patent No. 3,647,415 Yano,
et al., and which is an example of a tantalum flake that
is not produced in accordance with the teachings of that

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patent;

Figure 4 is a SEM at a magnification of 1000X, of a
flaked tantalum powder produced by subjecting the flake
5   of Figure 3 to the process of the present invention;

Figure 5 is a SEM at a magnification of 1000X, of a prior
art flaked tantalum powder produced in accordance with
Example I, specimen C, of the U.S. Patent No. 3,647,415
10   and which is representative of tantalum flake included
within the teaching of that patent;

Figure 6 is a SEM at a magnification of 500X, of a flaked
tantalum powder produced by subjecting the flake of
15   Figure 5 to the process of the invention;

Figure 7 is a SEM at a magnification of 1000X, of a
flaked tantalum powder produced in accordance with the
invention, and similar to that shown in Figures 2,4 and
20   except that the particles are smaller and about one-half
thickness; and

Figure 8 is a graph showing the particle size
distribution of the flaked tantalum powders shown in
25   Figures 3 to 6.

DETAILED DESCRIPTION OF THE INVENTION

Flaked tantalum powder may be prepared by deforming or
30   flattening a granular tantalum powder. It will be
appreciated by those skilled in the art that this
deformation may be accomplished by conventional
mechanical techniques using a ball mill, rod mill, roll
mill or the like. The flaked tantalum powder of the
35   present invention can be prepared from such
conventionally prepared flaked tantalum powder by
reducing the size of the flake particles until the mean

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particle size is in the preferred range of about 2 to
about 45 micrometers preferably about 3 to about 20
micrometers and BET surface area is in the range of about
0.5 to about 5.0 m^2/g, preferably, about 0.5 to about 5.0

5   m^2/g. The size reduction process may be aided by
embrittling the conventional flake by techniques such as
hydriding, oxidizing, cooling to low temperatures, or the
like, to enhance breakage when reducing the flake
particle size by mechanical means such as crushing, or

10   other size reduction processes. The mean particle size
and BET surface area are related, as hereinafter
described, and that relationship can be expressed as a
value, i.e., aspect ratio (D/T). Surprisingly, the BET
surface area may exceed 0.7 up to 5.0 m^2/g and is

15   preferrably inthe range of about 0.5 to about 1.5 m^2/g
without loss of the desired properties as long as mean
particle size is adjusted to maintain the aspect ratio
(D/T) in the range of about 2 to about 50 more
preferrably, about 5 to about 20.

20

In the present invention flakes are reduced in size
without substantial tapering of the flakes peripheral
edges. Consequently, in one embodiment the flaked
tantalum powder of the invention is characterized by

25   flakes of substantially uniform thickness from edge to
edge which are described herein as "fractured flake".
Thickness may be related to BET nitrogen surface area
values which are typically in the range of about 0.5 to
about 1.5 m^2/g. It should be kept in mind, however, that

30   the desired flake properties can also be achieved with
substantially thinner flakes (higher BET nitrogen surface
areas, e.g., greater that 1.5) if the mean particle size
is adjusted to maintain an aspect ratio at less than
about 50. Aspect ratios in the range of about 5 to about

35   20 being preferred.

Aspect ratios 'D/T' of flaked tantalum powders can be

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calculated from their mean particle size in micrometers
and BET (nitrogen) surface area in m^2/g in accordance with
the following formula:

$$D/T = (K) (MPS) (BET)$$

5   where: K   = 2, a constant;

MPS = mean particle size in
micrometers; and

BET = BET surface area (nitrogen).

The techniques for measuring MPS and BET are described in
10   greater detail below.

Comparison of Figures 1 through 9 illustrates that the
flaked tantalum powder of the present invention is
comprised of substantially smaller particles than the
15   prior art flakes. The prior art tantalum flake shown in
Figure 1 was prepared from classified 20 x 44 micrometer
ingot-derived (electron beam melted) tantalum chips. The
chips were degassed in a vacuum furnace to remove
hydrogen and sieved through a 325 mesh screen. The
20   resulting material was then milled in a vibrating ball
mill for 10 hours to flatten the chips into flake. This
flake was acid leached first in a HCl/HNO₃ mixture and
then in HF to remove metallic impurities. The resulting
flake which had a Scott density of 10.8 g/in³ was heat
25   treated at 1600° C for 30 minutes to produce an
agglomerated material which was then jaw crushed to 40
mesh sized agglomerates having a Scott density of 13.4
g/in³.

30   The flaked tantalum powder of the current invention shown
in Figure 2 was prepared from ingot-derived (electron
beam melted) 325 mesh tantalum chips. The chips were
degassed in a vacuum furnace to remove hydrogen and
sieved through a 325 mesh screen.   The resulting
35   material was milled in a vibrating ball mill for 10 hours
to flatten the chips into flake. This flake was acid
leached first in a HCl/HNO₃ mixture and then in HF to

10

remove metallic impurities. The resulting flake had a
BET nitrogen surface area value of 0.38 m²/g and a Scott
density in the range of 10 to 15.2 g/in³. This flake was
hydrided and subjected to cold isostatic pressing at

5   30,000 psi to break the flake into smaller pieces which
after pressing are in the form of a solid bar. The solid
bar was jaw crushed to 60 mesh producing flakes having a
BET nitrogen surface area value of 0.54 m²/g and Scott
density of 59.8 g/in³.

10

Comparison of the flaked tantalum powder shown in Figures
1 (prior art) and 2 (invention) demonstrates that the
flake of this invention is comprised of substantially
smaller flake particles.

15

The prior art flaked tantalum powder shown in Figure 3
was made from -60 mesh sodium reduced tantalum powder.
The powder was deformed to flake shape by milling in a
vibratory ball mill for ten hours. The ball milled

20   flakes were acid leached to remove metallic impurities
using 15% HCl and 2% HF. This method corresponds to the
procedure described in United States Patent No. 3,647,415
for preparing Specimen H in Example II. The Scott
density of the resultant flake was 12.54 g/in³ and 90% of

25   the flakes had no dimension larger than 126 micrometers
as shown in Table 1.

The flaked tantalum powder of this invention shown in
Figure 4 was made from -60 mesh sodium reduced tantalum

30   powder. The powder was deformed to flake shape by
milling in a vibratory ball mill for ten hours. The ball
milled flake was acid leached to remove metallic
impurities using 15% HCl and 2% HF. The flake was then
heated in a closed vessel until the flake reached about

35   850° C. Then, however, the heated tantalum flake was
hydrided by allowing it to cool to room temperature in
the vessel while a positive hydrogen pressure of +5 psi

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was maintained. The hydrided flake was reduced in size
by milling the flake material in a Vortec M1 impact mill,
available from Vortec Products Co., Long Beach
California, U.S.A., operating at 10,000 rpm. The

5   resultant flake had a Scott density of 21.45 g/in^3 and
about 90% of the flakes had no dimension greater than
about 37 micrometers.

The prior art flaked tantalum powder shown in Figure 5

10   was made from -60 mesh sodium reduced tantalum powder.
This powder had an absorbed hydrogen content of about 125
ppm. The powder was deformed to flake shape by milling
in a vibratory ball mill for six hours. Then the ball
milled flake was acid leached to remove impurities using

15   15% HCl and 2% HF. The resultant flake had a Scott
density of 12.7 g/in^3 and about 90% of the flakes had no
dimension greater than about 131.8 micrometers. This
method corresponds to the procedure described in United
States Patent No. 3,647,415 for preparing Specimen C in

20   Example I.

The flaked tantalum powder of this invention shown in
Figure 6 was made from -60 mesh sodium reduced tantalum
powder. The powder was deformed to flake shape by

25   milling in a vibratory ball mill for six hours. The ball
milled flake was acid leached to remove metallic
impurities using 15% HCl and 2% HF. The flake was then
heated in a closed vessel until the flake reached about
850° C. Then, however, the heated flake was hydrided by

30   allowing it to cool to room temperature in the vessel
while a positive hydrogen pressure of +5 psi was
maintained. The hydrided flake was reduced in size by
milling the flake material in a Vortec M1 impact mill
operating at 12,500 rpm. The resultant flake had a Scott

35   density of 28.30 g/in^3 and about 90% of the flakes had no
dimension greater than about 23.2 micrometers.

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The flaked tantalum powder of this invention shown in
Figure 7 was made from 100 mesh sodium reduced tantalum
powder. The powder was deformed to flake shape by
milling in an organic solvent using a Model 15S attritor
5   ball mill manufactured by Union Process Inc., Akron Ohio,
USA, for 16 hours to a BET nitrogen surface area greater
than about 1.0 m^2/gm indicating a thin flake. The ball
milled flake was acid leached to remove impurities using
15% HCl and 2% HF. The leached flake was then heated in
10 a closed vessel until the flake temperature reached about
850 °C, then cooled to room temperature and
hydrided/embrittled under hydrogen +5 psi. The hydrided
flake was further reduced in size by milling the flake
material in a Vortec M1 impact mill operating at 15,000
15 to 20,000 RPM. Higher RPMs are effective for fracturing
thinner embrittled flake to achieve the aspect ratios in
the desired range.

It is apparent from Figures 8 and 9 and the Granulometer
20 data in Table 1 that the particle size of the flake of
this invention is substantially smaller than the particle
size of the flake of the prior art. It also appears that
the particle size distribution of the flaked powder of
the invention is narrower than the particle size
25 distribution of the flake of the prior art.

The mean particle size, BET nitrogen surface area and
Scott density of the above-mentioned flaked powders may
be measured as described below.
30

Mean Particle Size
The mean particle size distribution of samples of flaked
tantalum powder are determined using a MICROTRAC II®
particle size analyzer available from the Leeds &
35 Northrup unit of General Signal Corporation, U.S.A. This
is an apparatus designed to measure the size of particles
suspended in a liquid via scattered light and laser

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optical methods. Particles between 0.34 and 60
micrometers are measured by the amount of scattered laser
light and particles between 0.12 and 0.34 by the amount
of scattered monochromatic light, impinging on a
5   photodetector array. By means of a self-contained
computer, the apparatus determines the distribution of
mean particle sizes. The Operator's Manual for the
MICROTRAC II® further describes the measurement method.

10   The sample numbers appearing in Table 1 (F3 through F6)
correspond to the flakes shown in Figures 3 through 6.
The measurements presented in Table 1 were made on a
Granulometer as indicated in the footnote. The results
of the Granulometer particle size distribution are set
15   forth in Table I and are graphed in Figure 8. In Figure
8, the solid lines reference the flaked tantalum powder
of the subject invention shown in Figures 4 and 6 and the
dotted lines reference the prior art flaked tantalum
powder shown in Figures 3 and 5.
20

A review of the data in Table 1 reveals that the particle
size of the flakes of the present invention are
substantially smaller than those of the prior art flakes,
which is consistent with comparative screen distribution
25   measurements. Further, the curves of Figures 8 and 9
show that the particle size distribution of the flakes of
the present invention are much narrower than those of the
prior art flakes.

30   BET Nitrogen Surface Area
The BET nitrogen surface area of flaked tantalum powder
samples can be measured with a MONOSORB® Surface Area
Analyzer available from QUANTACHROME Corporation,
Syosset, N.Y., U.S.A.. This is an apparatus designed to
35   measure the quantity of adsorbate nitrogen gas adsorbed
on a solid surface by sensing the change in thermal
conductivity of a flowing mixture of adsorbate and inert

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carrier gas such as helium. The methods and procedures
for making these measurements are described in the
instruction manual for the MONOSORB® apparatus. All BET
surface area measurements referenced herein were made
5 with nitrogen adsorbate gas.

Scott Density
Scott density is determined with an apparatus comprised
of a powder flowmeter funnel, a density cup and stand for
10 the funnel and cup available as a set from Alcan Aluminum
Corp., Elizabeth, N.J., U.S.A. The measurement is made
by pouring a flake sample through the funnel into the cup
(one-cubic-inch nickel plated) until the sample
completely fills and overflows the periphery of the cup.
15 Then the sample is leveled-off by means of a spatula,
without jarring, so that the sample is flush with the top
of the cup. The leveled sample is transferred to a
balance and weighed to the nearest 0.1 gram. The Scott
density is the weight per cubic inch of the sample.

15

TABLE I

Sample No.	F3 (Prior Art)	F4	F5 (Prior Art)	F6
Screen Distribution (% Sample retained)				
% +325	61.40	3.34	67.88	1.59
% -325/500	13.08	4.19	8.45	1.24
% -500	25.52	92.47	23.67	97.17
Scott Density				
(g/in³)	12.54	21.45	12.70	28.30
Granulometer Summary+				
(D) 10%*	33.5	6.7	41.6	4.6
(D) 50%**	83.5	17.4	93.2	11.1
(D) 90%***	126.5	37.0	130.8	23.2

+ As measured on Compagnie Industrielle Des Laser,
Cilas Alcatel Granulometer Model 715.

* Length in micrometers that is greater than the
measured diameter of the smallest 10 percent by volume
of the particles in the sample.

**Length in micrometers that is greater than the
measured diameter of the smallest 50 percent in volume
of the particles in the sample.

***Length in micrometers that is greater than the
measured diameter of the smallest 90 percent by volume
of the particles in the sample.

Agglomeration

The flaked tantalum powders are agglomerated in any
conventional manner in order to provide a product which
is suitable for subsequent formation into pellets from
which capacitor electrodes can be fabricated. Typically,
agglomeration involves heat treatment of the flake in a
vacuum or inert atmosphere at temperatures in the range
of about 1300 to 1600° C for periods of time ranging from
about 30 to 60 minutes. The specific agglomeration
technique utilized herein is described below.

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Agglomerates were made utilizing the flakes shown in
Figures 3, 4, 5 and 6. The flakes of Figures 3, 4, 5 and
6 were individually heat treated under vacuum at 1440° C
for 30 minutes and jaw crushed to -40 mesh. The flakes
5   were doped with 100 ppm phosphorus and then subjected to
a second heat treatment at 1500° C for 30 minutes and jaw
crushed to -40 mesh. The resulting flakes were
deoxidized by blending with 2.5% Mg powder and heating
under +3 psi Argon pressure to temperature of 950° C for
10   320 minutes. The resulting agglomerates were acid
leached, to remove MgO and excess Mg, using 15% HNO_3 and
then rinsed and dried.

Failure to flow is very detrimental since it is
15   impractical to commercially press pellets from
agglomerates that do not flow.

Pellet Fabrication and Crush Strength

20   An agglomerated flaked tantalum powder is compressed in a
conventional pellet press without the aid of binders
using an imbedded tantalum wire. Two samples of a
tantalum powder, one weighing 1.29 g and the other
weighing 1.33 g are separately introduced into a die of a
25   pellet press having a diameter of 0.250 inch. The press
is set up to press a pellet having a length of 0.330
inch. Utilizing the above weights and lengths an
approximate green density of 5.0 g/cc is achieved.

30   The flaked tantalum powder shown in Figure 7 was
agglomerated and pressed into pellets under conditions
similar to those described above. The resulting pellets
had a crush strength greater than 50 at 5.0 DP.

35   Although we have set forth certain present preferred
embodiments of our flaked tantalum powder and methods of
making same, it should be distinctly understood that our

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invention is not limited thereto but may be variously
embodied within the scope of the following claims.

We Claim:

1.   A method for making flaked tantalum powder that is flowable and pressable when agglomerated, comprising the steps of:

preparing tantalum flake powder;

reducing the flake size until the powder has a mean particle size in the range of about 2 to about 55 micrometers and BET surface area in the range of about 0.5 to about 5.0 m^2/g; and

selecting said mean particle size and BET surface area so that the reduced size flakes have an aspect ratio (D/T) in the range of about 2 to about 50.

2.   The method of claim 1, wherein the BET surface area of the reduced size flakes is greater than 0.70 m^2/g.

3.   The method of claims 1 or 2, wherein the flake size is reduced until the mean particle size is less than about 45 micrometers.

4.   The method of claims 1 or 2, wherein the flake size is reduced until the mean particle size is in the range of about 5 to about 20 micrometers.

5.   The method of claim 4, wherein the flake is prepared by a chemical reduction process.

6.   The method of claim 4, wherein the flake is ingot derived.

7.   The method of claim 1, wherein the aspect ratio (D/T) is in the range of about 5 to about 20.

8.   The method of claim 1, wherein the BET surface area of the reduced size flake is in the range of about 0.5 to about 1.5 m^2/g and the mean particle size is in the range of about 5 to about 20 micrometers.

9.   The method of claim 4, wherein at least about 90% of the reduced size flakes have no dimension greater than about 25 micrometers.

10. The method of claim 1 or 2, further comprising the step of embrittlement of the flaked tantalum powder prior to the size reduction step.

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11. The method of claim 4, further comprising the step of embrittlement of the flaked tantalum powder prior to the size reduction step.

12. The method of claim 10, further comprising the step of agglomerating the reduced size flake.

13. A low density pellet prepared from the reduced size flake of claim 4.

14. A capacitor prepared from the pellet of claim 13.

15. A flaked tantalum powder comprised of flakes having a mean particle size in the range of about 2 to about 55 micrometers and a BET surface area greater than about 0.7 m²/g; and an aspect ratio between about 2 and about 50.

16. The flaked tantalum powder of claim 15 wherein the tantalum is chemically reduced tantalum.

17. The flaked tantalum powder of claim 15 wherein the tantalum is ingot derived tantalum.

18. The flaked tantalum powder of claim 15 wherein the mean particle size of the reduced size flake is less than about 45 micrometers.

19. The flaked tantalum powder of claim 16, wherein the mean particle size of the reduced size flake is less than about 45 micrometers.

20. The flaked tantalum powder of claim 15, wherein the mean particle size of the reduced size flake is in the range of about 5 to about 20 micrometers.

21. The flaked tantalum powder of claim 20 wherein the BET surface area of the reduced size flake is in the range of about 0.5 to about 1.5 m²/g.

22. The flaked tantalum powder of claim 21 wherein the aspect ratio (D/T) of the reduced size flake is in the range of about 5 to about 20.

23. The flaked tantalum powder of claim 21, wherein the aspect ration (D/T) of the reduced size flake is in the range of about 5 to about 20.
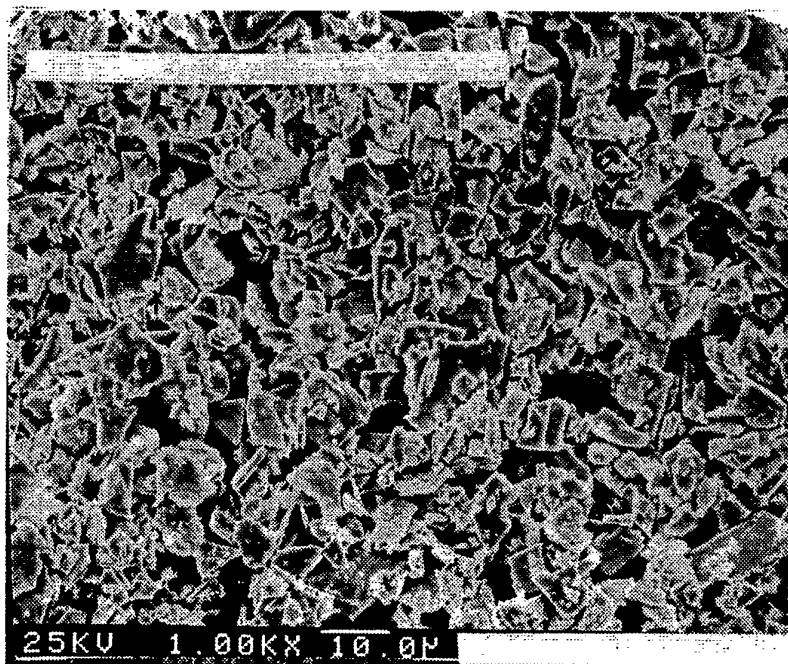
24. An electrode having low leakage and a high

20

breakdown voltage prepared with the powder of claim 15.

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FIG. 1
Conventional Tantalum Flake
1000X Magnification

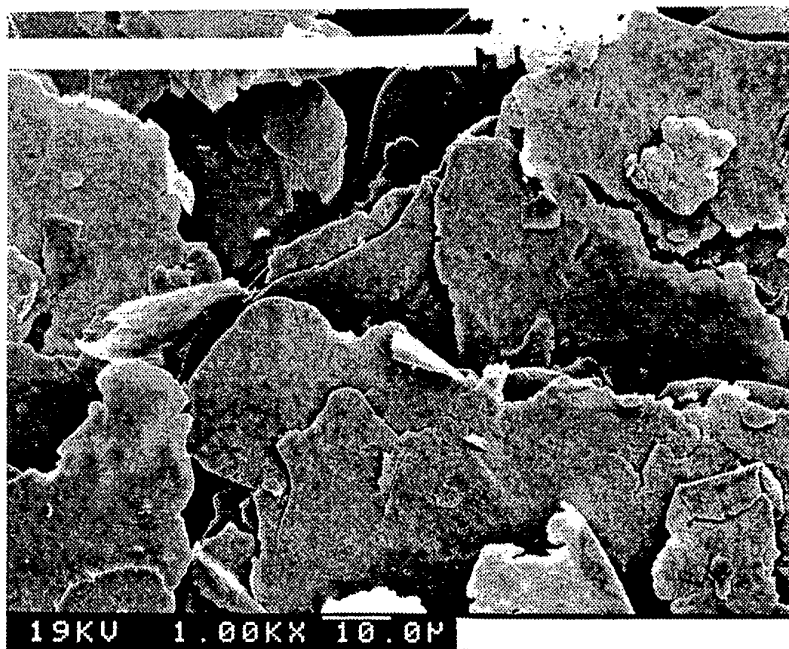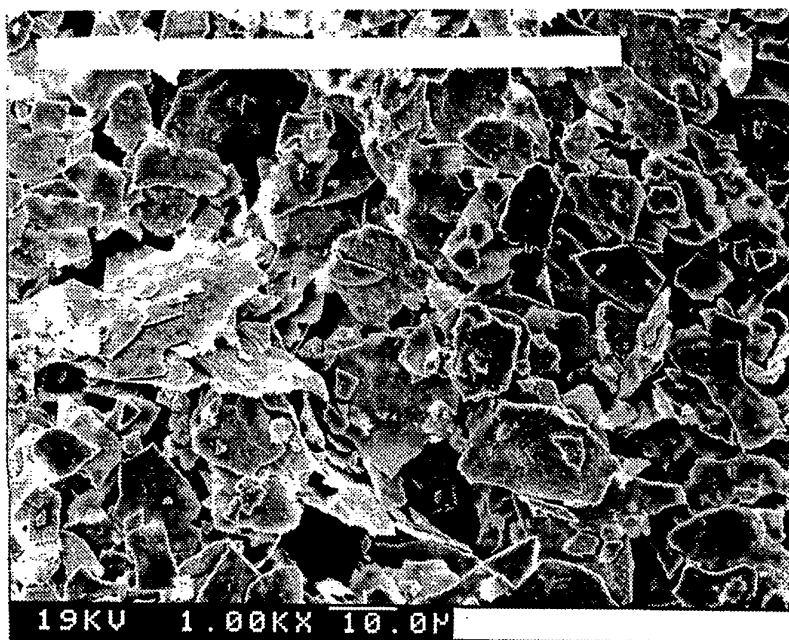# FIG. 2
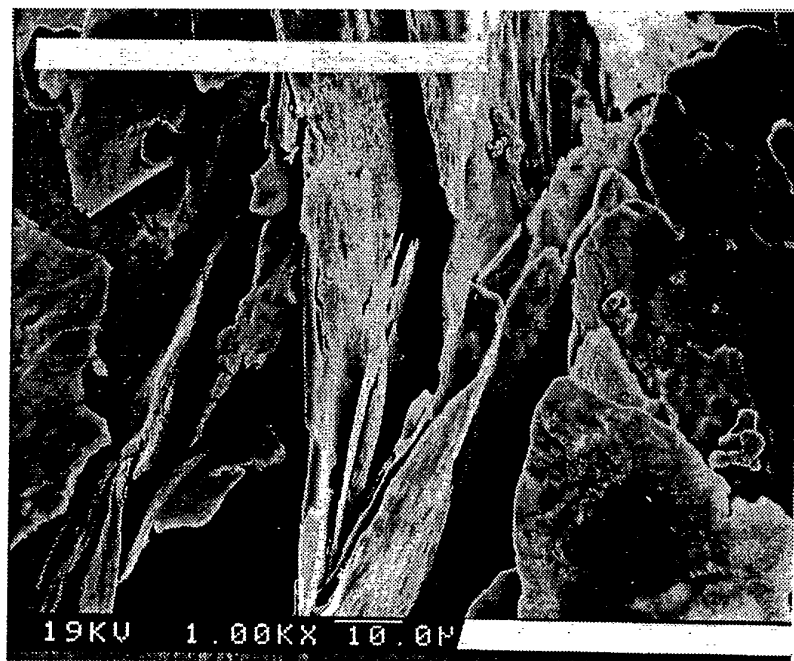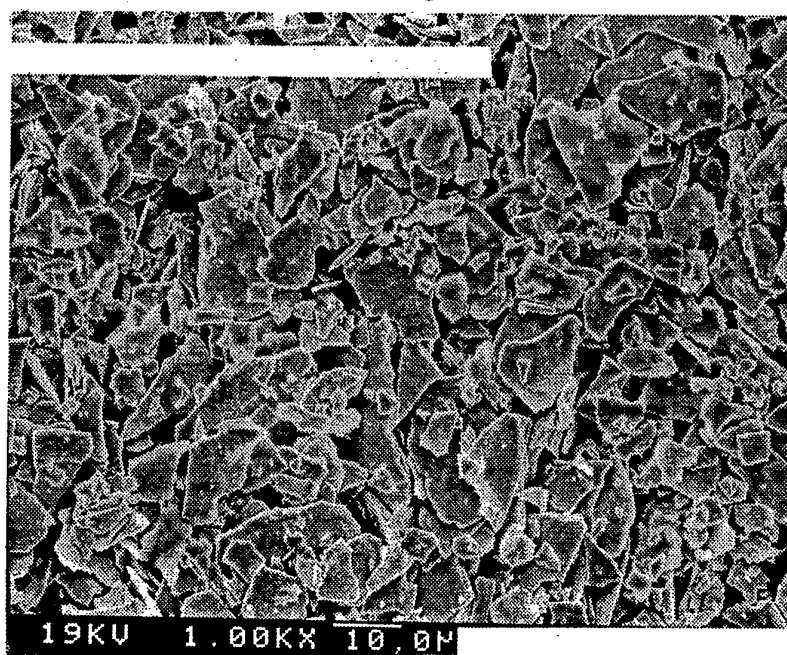Fractured Tantalum Flake
1000X Magnification

FIG. 3


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19KV    1.00KX  10.0M
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FIG. 4


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19KV    1.00KX  10.0M
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F I G. 5

19KU   1.00KX   10.0M

F I G. 6

19KU   1.00KX   10.0M

F I G. 7

19KV   1.00KX 10.0P 6330 5940-27-3
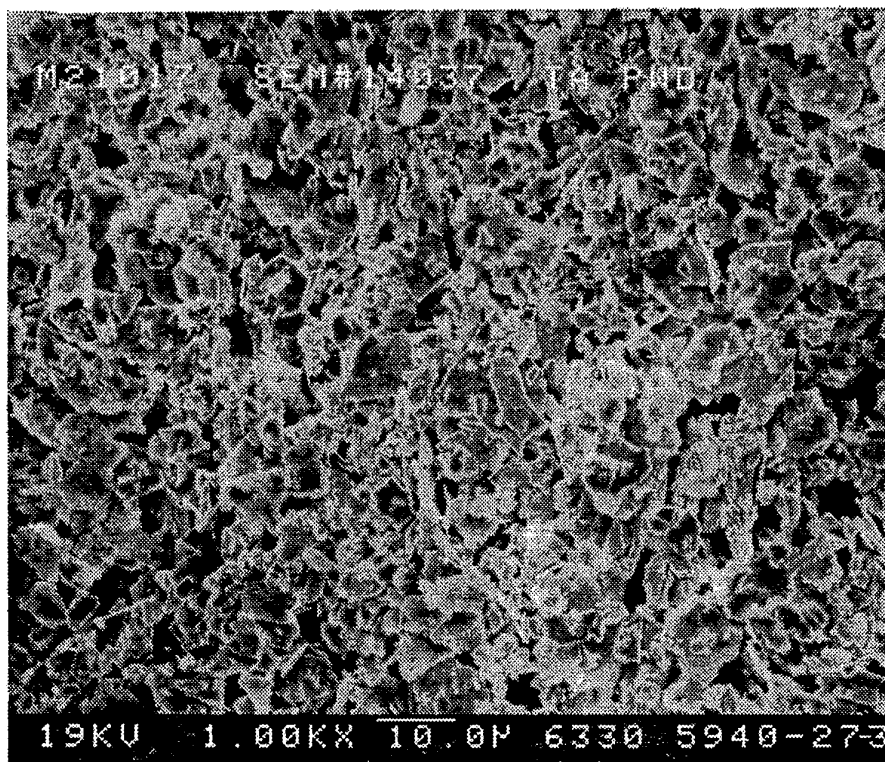
5/5

PARTICLE SIZE DISTRIBUTION
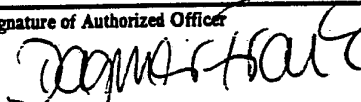
FIG. 8

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)[6]

According to International Patent Classification (IPC) or to both National Classification and IPC

Int.Cl. 5. C22C1/04;   H01G9/05

II. FIELDS SEARCHED

Minimum Documentation Searched[7]

Classification System	Classification Symbols
Int.Cl. 5	C22C ; H01G

Documentation Searched other than Minimum Documentation
to the Extent that such Documents are Included in the Fields Searched[8]

III. DOCUMENTS CONSIDERED TO BE RELEVANT[9]

Category°	Citation of Document,[11] with indication, where appropriate, of the relevant passages[12]	Relevant to Claim No.[13]
A	US,A,4 740 238 (SCHIELE) 26 April 1988 see column 1, line 5 - line 16; claims 1,2	1,13-15, 24
A	US,A,4 441 927 (GETZ ET AL.) 10 April 1984 see claim 1	1,13-15, 24
A	US,A,3 647 415 (YANO ET AL.) 7 March 1972 cited in the application	
A	DE,A,3 840 361 (CABOT CORP.) 8 June 1989	

° Special categories of cited documents:[10]

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

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"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
04 NOVEMBER 1992	2 0. 11. 92

International Searching Authority	Signature of Authorized Officer
EUROPEAN PATENT OFFICE	

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.
The members are as contained in the European Patent Office EDP file on
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information. 04/11/92

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