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D E C I S I O N
of 12 May 2003

Case Number: T 1020/99 - 3.2.2

Application Number: 94306631.6

Publication Number: 0648854

IPC: C22C 38/60

Language of the proceedings: EN

Title of invention:
Martensitic hot work tool steel die block article and method of manufacture

Applicant:
CRUCIBLE MATERIALS CORPORATION

Opponent:
-

Headword:
-

Relevant legal provisions:
EPC Art. 52(1), 56

Keyword:
"Inventive step (no)"

Decisions cited:
-

Catchword:
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Case Number: T 1020/99 - 3.2.2

D E C I S I O N
of the Technical Board of Appeal 3.2.2
of 12 May 2003

Appellant: CRUCIBLE MATERIALS CORPORATION
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Decision under appeal: **Decision of the Examining Division of the European Patent Office posted 1 June 1999 refusing European patent application No. 94 306 631.6 pursuant to Article 97(1) EPC.**

Composition of the Board:

Chairman: W. D. Weiß
Members: S. S. Chowdhury
U. J. Tronser

Summary of Facts and Submissions

I. This appeal is against the decision of the examining division dated 1 June 1999 to refuse European patent application No. 94 306 631.6.

The ground of refusal was that, having regard to the following documents cited by the examining division, the subject-matter of claim 1 lacked an inventive step:

D1: J. R. Davies et al., Metals Handbook, 10th edition, Vol. 1, pages 780 to 792, 1990

D2: JP-A-61 130 467

D3: US-A-3 598 567

D4: J. R. Davies et al., Metals Handbook, 9th edition, Vol. 16, pages 733 to 735, 1989.

The examining division argued that, starting from document D1, the problem to be solved was to improve the machinability of hot-work tool steels, and that it would be obvious to the person skilled in the art to add sulfur to hot-work steels to improve their machinability given that the concept of adding sulfur to steels produced by conventional ingot casting to improve machinability was well known for many years. D4 made the person skilled in the art aware that sulfur could be added to P/M (powder metallurgy) tool steels in general to improve machinability without impairing toughness, and it would, therefore, take no imagination to add sulfur to hot-work tool steels.

II. On 19 July 1999 the appellant (applicant) lodged an appeal against the decision and paid the prescribed fee on 22 July 1999. On 22 September 1999 a statement of grounds of appeal was filed.

With the grounds of appeal the appellant cited the following documents in support of his arguments:

D5: "The effects of Sulfur Content on the Performance of H-13 Steel", Du et al., 1983

D7A: Declaration of Kenneth E. Pinnow, PhD

D8: "Thermal Fatigue Test Results for Commercial Hot-work Tool Steels", Crucible Research internal report dated November 1988.

The Board has also considered the following further document which was cited *ex officio*:

D9: EP-A-0 249 855

and the following further documents cited by the appellant in the ongoing course of the appeal procedure:

D12: Decision of the EPO Technical Board of Appeal, T 1021/99

D13: "Influence of Slag Particles on the Mechanical Properties of a P/M High Speed Steel" -Ambeig and Karlsson, International Journal of Powder Metallurgy, Volume 24, No. 3, 1988

D14: "Effect of Volume Fraction and Shape of Sulfide Inclusions on Through-thickness Ductility and Impact Energy of High-Strength 4340 Plate Steels" -Speich and Spitzig, Metallurgical Transactions A, Volume 13A, December 1982, pages 2230 to 2258.

III. Following a communication from the Board inviting the appellant to oral proceedings, the appellant's representative filed new claims on 10 March 2003 as an auxiliary request, and withdrew its earlier request for oral proceedings.

IV. The appellant requests that the decision under appeal be set aside and that a patent be granted on the basis of the following claims:

Main request

Claims 1 to 4, 6 (part) and 7 to 10 received on 15 January 1998 with letter dated 14 January 1998.

Claims 5, 6 (part) received on 2 July 1998 with letter dated 1 July 1998.

Auxiliary request

Claims 1 to 10 filed by telefax dated 10 March 2003.

V. Independent claim 1 of the main request reads as follows:

"A martensitic hot-work tool steel die block article adapted for use in the manufacture of die casting die components and other hot-work tooling components, said article having a hardness within the range of 35 to 50

HRC, and a minimum transverse Charpy V-notch impact toughness of 7 J (5 foot-pounds) when heat treated to a hardness of 44 to 46 HRC and when tested at both 22°C (72°F) and at 316°C (600°F), said article comprising a hot-worked heat treated and fully dense consolidated martensitic hot-work tool steel mass of prealloyed particles having 0.05 to 0.30 weight-percent sulfur and having sulfide particles with a maximum size of 50 micrometers in their longest dimension.".

This request also contains independent claims 2 and 3 directed to a martensitic hot-work tool steel die block article, and independent claim 4 directed to a martensitic steel die article, and independent claims method claims 6 to 8 directed to methods of manufacturing such articles, but for the present decision only claim 1 of the main and auxiliary requests is relevant.

Independent claim 1 of the auxiliary request has the same wording as claim 1 of the main request, except that the words "gas atomised" have been added before "particles" in the third last line of the claim.

VI. The appellant argued as follows:

High-S speciality tool steel grades were available as early as 1973, despite which the authors of D1 deliberately separated hot-work tool steel compositions from the other steel compositions in Table 1. This was because there were severe difficulties in producing highly sulfurised P/M hot-work tool steel grades that exhibited the desired physical characteristics claimed in claim 1.

This argument was supported by D13 which clearly stated that both high N and S contents degrade the impact energy and bend fracture strength of the high speed steel alloys under consideration. The person skilled in the P/M art would have understood that hot-work tool steels could not be manufactured in highly resulfurised grades using P/M techniques because of the tendency of high S-contents to degrade the impact energy and bend fracture strength, which was of no consequence for high speed tool steels. D13 also expressed a clear prejudice against adding high sulfur contents to hot-work tool steels. D14 explained why the Charpy impact shelf strength fell in AISI 4340 steel in high-sulfur compositions unless rare earth elements were added.

The decision D12 failed to address the fact that D3 disclosed no more than powders, and not the hot-work tool steel article. During hot-working the interstitial or inter-particle sulfides would elongate, but if the sulfide size were maintained to be less than 50 µm, according to the application, then the deleterious effect would be avoided.

Reasons for the Decision

1. The appeal is admissible.

The following arguments pertain to the auxiliary request since the arguments apply equally to the main request. The reason for this is that gas atomisation is the preferred and most widely used method of atomising particles in the P/M process, as discussed in point 3.1, below, and does not materially alter the scope of the claim.

2. *Novelty*

Novelty of the claimed subject-matter was not doubted by the examining division. The Board agrees with this as will emerge from the discussion of inventive step, below.

3. *Inventive step*

3.1 Technical background, the closest prior art

D1 describes tool steels produced by powder metallurgy (P/M), and contains the following different sections: A first, general section describing P/M tool steels, a second section dealing with P/M high-speed tool steels, a third section dealing with P/M cold-work tool steels, and a fourth section dealing with P/M hot-work tool steels. The basic production process described in D1 produces a fully dense consolidated mass of water or gas atomisation of prealloyed steel particles, (page 780, middle column), and the compacted mass is hot-worked (page 780, right column).

The fourth section, commencing on page 789 and dealing with P/M hot-work tool steels, mentions their use for die applications, and Table 1 on page 781 gives the compositions of three of hot-work tool steels. As an example, the mechanical properties of the martensitic steel type P/M H13 after a standard heat treatment (1010°C/1h - air cool - 593°C/2+2h; cf. D1, page 789, column 3, 2nd complete paragraph) are given in D1, Tables 8 and 10 (hardness: 47.5-48.1 HRC and toughness: Charpy V-notch impact strength: 10. ft-lbf). However, Table 1 and the section "Hot-Work Tool Steels" do not explicitly mention powder metallurgy processing of

hot-work tool steels to which increased amounts of sulfur were added in order to improve machinability.

3.2 Technical problem

The object of the application is to provide a highly machinable, prehardened, martensitic hot-work tool steel die block which contains intentional additions of sulfur and which may be used to manufacture die casting die components and other hot-work tooling components having an improved combination of impact toughness, machinability, and thermal fatigue resistance.

This object is achieved by adding 0.05 to 0.30 wt% sulfur and ensuring that the sulfide particles have a maximum size of 50 µm in their longest dimension.

3.3 In light of the above technical background, it is, therefore, necessary to adjudicate on whether it was obvious to a skilled person to use the P/M route for producing hot-work tooling components consisting of resulfurized grades of hot-work tool steel having sulfide particles with a maximum size of 50 µm in their longest dimension with the expectation of obtaining improved impact toughness, machinability, and thermal fatigue resistance.

3.4 As set out in the application (A1 publication) on page 2, lines 24 to 28, prehardened die blocks made from ingot metallurgy (I/M) resulfurized H-13 steel were known in the art. Whilst improving machinability, the increased sulfur content entails the drawback of reducing the thermal fatigue resistance and impact toughness of the I/M-steel, which are required for good die performance and die service life. This degradation

of the mechanical properties in ingot metallurgy is caused by segregations of sulfur which form a non-uniform distribution of numerous sulfides of different morphology.

The chapter "P/M Hot-Work Tool Steels" on page 789 of document D1 also states that a frequent cause of premature failure of large die casting dies is thermal fatigue which is attributed to segregations and a heterogeneous microstructure. D1 goes on to say that P/M processing offers an alternative method of producing segregation-free hot-work tool steels of both standard and improved compositions and further offers near net shape capability (cf. D1, page 789, second column). It is, therefore, the powder metallurgy route which provides the metallurgist an encouraging prospect of overcoming the drawbacks associated with segregation phenomena in general and sulfur segregation in particular.

It was also known from document D3 that cooled ingots suffer from the formation of coarse secondary phases, dendrites, aggregates, etc., such as carbides, sulfides, etc., which leads to poor physical properties such as fatigue and impact resistance (column 2, lines 14 to 26), and proposes to atomize steel alloys containing substantial amounts of phase forming constituents, especially sulfur, which form segregatable phases in I/M (cf. D3, column 1, lines 33 to 39; column 2, lines 8 to 55; column 4, lines 53 to 55; Example 6). More specifically, document D3 mentions (column 8, lines 62 to 69) the production of alloys capable of being made free-machining including tool steels and hot-work die steels such as those referred to in the trade as 4130, 52100, and Cr-Mo

steels comprising 5% Cr, 1% Mo, 0.55% V, 0.5% C and the balance being iron. The metal powder produced by atomizing can be hot-worked by hot consolidation (column 3, lines 32 to 38).

3.5 Therefore, while sulfur was not added to hot-work tool steels produced by ingot metallurgy because of segregation-related problems, there was sufficient incentive in the prior art to add sulfur to hot-work tool steels produced by powder metallurgy in order to improve toughness and thermal fatigue life because the P/M route avoids segregation. The application simply uses this teaching since the superior impact toughness of the claimed article also originates from a segregation-free microstructure and by avoiding growth of the sulfides, by taking advantage of P/M technology.

3.6 As regards the size of the sulfides, the prior art makes it clear that the benefits of powder metallurgy accrue largely due to the very fine microstructure with a uniform distribution of carbides and nonmetallic inclusions (D1, first column on page 780, second paragraph). Moreover, D1 (page 782, left column) mentions the small size of the sulfides as a distinguishing feature, and it is stated on page 734 of D4, middle paragraph, that "Because of the small size and uniform distribution of the sulfides, more sulfur (with a corresponding greater improvement in machinability) can be used in P/M tool steels than in conventional tool steels before hot workability or mechanical properties are degraded". The sulfide inclusions produced in typical P/M processes have a size of the order of microns (see, for example, D1, page 782, first complete paragraph and Figure 2(b) which explains what is meant by "small", ie the sulfide

size is about 8 µm, and D3, column 5, lines 23 to 27, D13, page 204, Results). In D3 the sulfide inclusions are said to be less than 2 microns in size when wet steam atomisation was used (column 5, lines 23 to 27), but gas atomisation would produce a comparable sulfide size.

Therefore, the prior art contains ample teaching that in order to obtain good mechanical properties growth of the sulfides should be avoided. It follows that in order to improve the mechanical properties of a P/M tool steel it is necessary to ensure that the sulfides are not excessively elongated, otherwise there is the risk of undoing the benefits of small sulfide size attained by the P/M route. In any case, the sulfide size after HIPing and hot-working would not exceed 50 µm even no precaution were taken in this respect, particularly when near net shapes are produced before HIPing.

3.7 The Board has taken into account the Declaration of Dr Pinnow (D7A), but is still convinced that it cannot follow the appellant's evaluation of the contents of document D1. Despite the possible negative side effects, resulfurized hot-work tool steels for the claimed purpose have already been produced in the art, and there is nothing in the standard textbook D1 which the skilled reader would interpret as a serious prejudice which had to be overcome when adding sulfur to the steel grades under consideration.

The first section of D1, starting on page 780, describing P/M tool steels in general, does not explicitly teach against the addition of sulfur to P/M hot-work tool steels. Table 1 and the fourth section

starting on page 789, while not disclosing hot-work tool steels with added sulfur, also do not explicitly teach against the addition of sulfur to P/M hot-work tool steels. D3, on the other hand, expressly teaches the addition of sulfur to hot-work PM tool steels in order to improve machinability.

The appellant's argument, that D13 also expressed a clear prejudice against adding high sulfur contents to hot-work tool steels, is also not accepted by the Board. This document states that sulfur degrades the impact energy and bend fracture strength of a P/M high-speed steel, and is, therefore, less relevant than the teaching in D1 regarding degradation of the toughness and thermal fatigue life of hot-work steels owing to segregations.

In order to establish the existence of a technical prejudice against the performance of a certain action, the solution must clearly clash with the prevailing teaching of experts in the field. The appellant has not clearly demonstrated that it was generally accepted that sulfur would not normally be added to hot-work tool steels.

3.8 The above opinion of the disclosure of document D1 is not changed by the technical results presented in document D5. All the tests in this document were performed on specimens which were produced by melting the alloy in a high frequency induction furnace and casting an ingot. This represents the typical I/M route and the products suffer from the drawbacks associated therewith. The same statement is true for document D8 which discloses thermal fatigue test results for commercial (I/M) hot-work tool steels comprising sulfur

in the range of 0.001 to 0.021%, and D14 also reports the effect of sulfide inclusions in steel produced by the I/M route.

3.9 The appellant has also criticised the decision T 1021/99 (D12) in that it failed to address the fact that D3 disclosed no more than powders, and not the hot-work tool steel article. As stated in D1 (see page 789, last paragraph), articles produced by the P/M route have a near net shape before HIPing, so no appreciable growth or distortion of sulfide inclusions is expected after HIPing during hot-working. There will be some growth during the HIPing process, but the growth would not be of the order of a magnitude. Moreover, as argued in point 3.6 above the person skilled in the art would take precautions against inordinate growth of the sulfides.

3.10 In view of these considerations, the subject matter of claim 1 of the auxiliary request, and therefore also of the main request, does not involve an inventive step.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

V. Commare W. D. Weiß