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D E C I S I O N
of 17 December 2002

Case Number: T 1021/99 - 3.2.2

Application Number: 94306633.2

Publication Number: 0648852

IPC: C22C 33/02

Language of the proceedings: EN

Title of invention:

Hot-isostatically-compacted martensitic steel article for
molds and die components and its method of manufacture

Applicant:

CRUBICLE MATERIALS CORPORATION

Opponent:

-

Headword:

-

Relevant legal provisions:

EPC Art. 56

Keyword:

"Inventive step (no)"

Decisions cited:

-

Catchword:

-



Case Number: T 1021/99 - 3.2.2

D E C I S I O N
of the Technical Board of Appeal 3.2.2
of 17 December 2002

Appellant: CRUBICLE 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. 94 306 633.2 pursuant to Article 97(1) EPC.

Composition of the Board:

Chairman: W. D. Weiß
Members: R. Ries
U. J. Tronser

Summary of Facts and Submissions

I. The present appeal is against the decision of the Examining Division to refuse European patent application 94 306 633.2 (EP-A-0 648 852) for lack of inventive step. Reference was made in the decision *inter alia* to the following documents:

D1: Metals Handbook, Tenth Edition, volume 1, March 1990, pages 780 to 792

D3: US-A-3 598 567

In the decision under appeal, the Examining Division held that document D1 discloses the production of resulfurized grades of P/M **high speed** tool steels but does not expressly mention a sulfur content of 0.05 to 0.30% in P/M **hot-work** tool steels. Nevertheless, it was found that document D1 teaches the well-known concept of adding sulphur in much higher than conventional levels to P/M tool steels **in general** (ie. high speed steels, cold work tool steels and hot-work steels produced by the powder metallurgical route) to improve their machinability without sacrificing toughness or cutting performance. Since in the P/M process, the rapid solidification of the atomized powders eliminates in the particles the segregations of sulphur which deteriorate toughness and the fatigue properties, the P/M tool steel compacts exhibit a very fine microstructure with a uniform distribution of carbide and non-metallic inclusions (such as sulfide particles). Based on the technical background reflected by document D1, the skilled person is made aware that sulphur can be added to tool steels in general without impairing toughness, and, consequently, it was obvious

to add sulphur also to P/M **hot work** steel to solve the problem of increasing the machinability of these grades.

II. In its notice of appeal, the appellant (patent applicant) referred to

D5: "The Effects of Sulphur content on the Performance of H13 Steel", Du et al., published in 1983, The Die Casting Research Foundation, paper No. 01-83-01D, American Die Casting Institute Inc.,

D6: E-mail from Ms Pia Björk, dated 15 January 1999

D7: Declaration of Dr Pinnow (D/a) plus curriculum vitae (D7b)

D8: "Thermal Fatigue Test Results for Commercial Hot Work Tool Steels", Crucible Research Internal Report dated November 1988

and requested that the decision under appeal be set aside and that a patent be granted on the basis of the unamended documents. No subsidiary requests were submitted. On an auxiliary basis, the appellant requested oral proceedings.

III. The Board issued a summons to oral proceedings expressing doubts on the appellant's view that the technical teaching given in document D1 would have prevented a person skilled in the art from adding sulphur also to P/M **hot-work** tool steels. In this context the Board additionally referred to the documents

D9: EP-A-0 249 855

D10: US-A-3 723 094.

showing that resulphurized grades of hot work steel (such as H-13S) had already been produced by the ingot metallurgy (I/M) and powder metallurgy (P/M) route.

IV. In its letter dated 11 September 2002 in response to the summons to oral proceedings, the appellant informed the Board that no further submissions in the case would be made and that the request for oral proceedings was withdrawn. Moreover, the Appeal Board was requested to make a decision on the basis of file as it stood.

Independent claim 1 underlying the appealed decision reads as follows:

"1. A martensitic hot work tool steel mold and die block article adapted for use in the manufacture of molds for plastic injection molding, die casting die components, and other hot work tooling components, said article having a hardness within the range of 35 to 50 HRC, a minimum Charpy V-notch impact toughness of 4 J (3 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 an as hot-isostatically-compacted, fully dense, heat treated mass of prealloyed particles, which contains sulfur within the range of 0.05 to 0.30 weight percent."

Independent product claims 2 to 4 include all the features of claim 1 and further define the composition of the prealloyed particles (in bold letters):

"2. A martensitic hot work tool steel mold and die block article mass of prealloyed particles comprising, in weight percent, 0.32 to 0.45 carbon, 0.20 to 2.00 manganese, 0.05 to 0.30 sulfur, up to 0.03 phosphorus, 0.80 to 1.20 silicon, 4.7 to 5.70 chromium, 1.10 to 1.75 molybdenum, 0.80 to 1.20 vanadium, up to 2.00 niobium, balance iron and incidental impurities"

"3. A **hot-isostatically-compacted** martensitic hot work tool steel mold and die block article mass of prealloyed particles comprising a chemical composition of any of AISI hot work tool steel to which sulfur has been added within the range of 0.05 to 0.30 weight percent."

"4. A **hot-isostatically-compacted** martensitic hot work tool steel mold and die block article..... mass of prealloyed particles comprising a chemical composition of a maraging or precipitation-hardening steel which is suitable for use as molds for plastic injection molding, die casting die components, and other hot work tooling components and to which sulfur has been added within the range of 0.05 to 0.30 weight percent."

Independent method claim 8 reads:

"8. A method for manufacturing 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, the article having a hardness within the range of 35 to 50 HRC and a minimum transverse Charpy V-notch impact toughness of 4 J (3 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 an as hot-isostatically-compacted, heat treated and fully dense consolidated mass of prealloyed particles comprising, in weight percent, 0.32 to 0.45 carbon, 0.20 to 2.00 manganese, 0.05 to 0.30 sulfur, up to 0.03 phosphorus, 0.80 to 1.20 silicon, 4.75 to 5.70 chromium, 1.10 to 1.75 molybdenum, 0.80 to 1.20 vanadium, balance iron and incidental impurities;

said method comprising producing said prealloyed particles by gas atomization, hot isostatically compacting the prealloyed particles to full density to form a compact and absent of thermomechanical treatment of said compact, annealing said compact, hardening said compact by heating and cooling to produce a martensitic structure, and tempering said compact, which tempering includes at least a double tempering treatment with intermediate cooling to ambient temperature."

Independent claims 9 and 10 differ from claim 8 in that the prealloyed particles

- "comprise a chemical composition of wrought AISI hot work tool steel to which sulfur has been added within the range of 0.05 to 0.30 weight percent" (claim 9) or
- " comprise a chemical composition of a maraging or precipitation-hardening steel suitable for... to which sulfur has been added within the range of 0.05 to 0.30 weight percent... and age hardening said article to working hardness by heat treating and cooling" (claim 10).

V. In the written proceedings, the appellant argued as

follows:

The problem underlying the present application was to provide a hot work P/M tool steel which exhibited (a) an improved machinability in combination with (b) a good thermal fatigue resistance and (c) without impairing the toughness of the steel.

The application solved this problem by hot isostatic pressing (HIPping) the resulphurized P/M hot work tool steel article to full density and heat treating the article. The improved mechanical performance and properties of the hot work steel article are shown in Figures 4, 5, 6b and 6c.

As disclosed in document D1, it was known to manufacture hot work tool steels using the P/M technique before the priority date of the application. However, document D1 does not provide any motivation to add sulphur to P/M hot work tool steels as alleged by the examining division. On the contrary, all references in D1 to the addition of sulphur are exclusively made in the context of P/M **high speed** tool steels, and the only category of steel to which sulphur is **not** added is that of P/M hot work tool steels. The skilled reader's perception of document D1 would, therefore, prevent him, in the light of his technical knowledge, from adding sulphur to hot work tool steels since doing this would markedly impair the mechanical properties rather than improve them. This evaluation of the contents of document D1 is corroborated by the Declaration of Dr Pinnow (document D7a) who was one of the authors of document D1. According to Dr Pinnow, D1 makes a clear and correct distinction between hot work tool steels (to which sulphur is not added) and cold work tool

steels (to which sulphur can be added) and, therefore, this document does not comprise any example of a resulphurized hot work tool steel. According to Dr Pinnow (cf. D7), it was believed that in hot-work tool steels increased sulphur contents adversely affect the toughness and the thermal fatigue life of articles made from these steels. This is confirmed by document D5 which deals with the effect of the sulfur content on the performance of H13 steel. It is found that S-contents up to 0.028% have little influence on the thermal fatigue resistance, but a sulfur content up to 0.075% results in a marked decrease of thermal fatigue resistance. Larger amounts of sulfide inclusions are found to be brittle second phases which reduce the hot yield strength and fracture toughness by increasing the concentration of thermal stress (see D5, page 14).

It is therefore concluded that, although the skilled man knew about the beneficial effect of sulphur additions on the machinability of P/M high speed steels, the addition of sulphur to hot work tool steels was not envisaged since increased contents of sulphur were expected to degrade toughness and the resistance to thermal fatigue, in particular, if the steels were subject to regular thermal cycling as occurs in die casting applications (cf. document D8).

The technical teaching given in documents D2 (no disclosure of hot work tool steels) and D3 is irrelevant, in particular since document D3 only relates to the composition of powders and not to a P/M article as claimed in the present application.

Moreover, a fair chance was not given to the applicant

to comment on the ground of refusal. Given that the Examining Division's summons to oral proceedings dated 23 March 1999 did not comment on the primary examiner's positive reasoning given in an earlier communication (D6: e-mail dated 15 January 1999) and indicating likely allowability of the application, the applicant assumed that the comments in the e-mail continued to apply. The applicant was, therefore, detracted from its ability to concentrate on the points mentioned in the communication of the Examining Division dated 23 March 1999. The Examining Division, therefore, did not satisfy the common provisions governing procedures pursuant to Article 113(1) EPC.

Reasons for the Decision

1. The appeal is admissible.

2. *Technical background, the closest prior art*

The technical background to the present application is amply reflected by the chapter "P/M Tool Steels" given in document D1 which is a Standard text book in the field of metallurgy and represents the closest prior art. As set out in the introductory part of D1 on page 780, first column, the P/M tool steels (in general) offer several distinct advantages over conventional tool steels produced by I/M, including a very fine microstructure with a uniform distribution of carbides and non-metallic inclusions and the absence of segregations. With particular respect to "hot-work tool steels", the P/M route is said to offer a greater toughness and a better thermal fatigue life. More specifically, the section "P/M Hot-Work Tool Steels"

starting on page 789 discloses the P/M route to be an alternative method of producing segregation-free hot-work tool steels of both standard and improved compositions and offering the ability to produce near-net shape die cavities directly during HIPping which minimizes material input and subsequent machining (cf. D1, page 789, last paragraph of columns 1, 2 and 3). Since a fully dense porous free structure (100% density) of the article is achieved after HIPping, there is no need for an additional thermomechanical treatment such as hot forging, hot rolling or hot extrusion (cf. D1, page 780, column 2, lines 4 to 8). As confirmed by document D1, page 790, column 3, lines 1 to 7, the die is after HIPping ready for heat treatment and finish machining, which means that no further thermomechanical treatment changing the microstructure after HIPping is envisaged in this process. 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 full 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 on page 781 and the section "Hot-Work Tool Steels" of document D1 do not explicitly mention the powder metallurgy processing of hot-work tool steels to which increased amounts of sulphur were added in order to improve their machinability.

3. *Inventive step*

Before this technical background, it is, therefore, necessary to consider 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 exhibiting an improved machinability without sacrificing the notch toughness, fatigue life and degrading the polishability.

- 3.1 As set out in the application (A1 publication) on page 2, lines 20 to 28, prehardened mold and die blocks made from an **I/M** resulfurized H-13 steel are known in the art. Whilst improving the machinability, the increased sulphur content entails the drawback of reducing the notch toughness and degrading the polishability of the I/M-steel which are required for plastic injection molding applications. This degradation of the mechanical properties in the **ingot** metallurgy is caused by the segregations of sulphur which form a non-uniform distribution of numerous sulfides of different morphology. One approach to control these segregations of sulphur within reasonable limits is disclosed in document D10 which suggests the production of a hot-work die steel of resulfurized grade **H-13S** by electroslag remelting to provide a more uniform sulphur distribution from the bottom to the top in the final ingot (cf. D10, column 1, lines 23 to 27, Example 2, S = 0.121%). Another hot-work tool (die) steel produced by the I/M route and having physical properties (hardness, strength and toughness) at least comparable to those of standard type H13 is disclosed in document D9, page 2, Summary of the invention and page 5, lines 1 to 35. If desired, this hot-work tool steel composition can further include free machining additives such as up to 0.10% sulphur (cf. D9, page 2, last paragraph; page 5, first paragraph).
- 3.2 As an alternative method to ingot metallurgy, document D3 proposes the atomization of steel alloys

containing substantial amounts of phase forming constituents (especially sulphur, which in I/M form segregatable phases) and hot consolidating the powder mass (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 in 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. Consequently, the production of resulphurized grades of hot work tool steels either by the I/M or by the P/M route was known in the art.

- 3.3 Even after taking into account the Declaration of Dr Pinnow, the Board cannot follow the appellant's evaluation of the contents of document D1. Despite the possible negative side effects, resulphurized 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 in the skilled reader's perception could be interpreted as a serious prejudice which had to be overcome when adding sulphur to the steel grades under consideration. On the contrary, the chapter "P/M Hot-Work Tool Steels" on page 789 of document D1 clearly 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 to overcome the drawbacks associated with segregation phenomena in general and sulphur segregation in particular (cf. also document D3). Also the superior impact toughness and polishability of the mold and die block claimed in the present application originate from a segregation-free microstructure and the small spherical shape and more uniform distribution of the sulfides by taking advantage of the P/M technology.

This evaluation of the contents of document D1 cannot be changed by the technical results presented in document D5. All 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%.

In view of these considerations, the subject matter of claim 1 does not involve an inventive step.

- 3.4 The same reasoning is true for independent claims 2, 3 and 4 which are directed to P/M mold and die articles produced from different compositions of well known hot work tool steels.

- 3.5 Compared with the product claims 1 to 4, independent method claims 8 to 10 additionally comprise the typical standard heat treatment and processing steps which are disclosed for example in document D1, page 789, column 3, second full paragraph. Hence, also the

claims 8 to 10 do not comprise technical features justifying an inventive step.

4. *Procedural matters*

In the course of the proceedings before the Examining Division, the appellant (applicant) was informed via a telephone conversation with the formalities officer on 22 February 1999 that the first examiner entrusted with the substantive examination of the file had departed and, therefore, the members forming the Examining Division needed to be changed. In order to meet the applicant's request submitted on 1 March 1999 for expediting the prosecution of the application, the members forming the new Examining Division immediately prepared for oral proceedings which took place on 22 April 1999. In the official communication accompanying the summon for oral proceedings, the Examining Division referred explicitly to the most relevant prior art, ie. the technical teaching given in standard textbook D1, and expressed serious doubts as to the inventive step of the subject matter of claim 1. It should, therefore, have been clear to the applicant that the members of the new Examining Division were disinclined to accept the positive view expressed by the former primary examiner. In response and enclosed with its letter dated 20 April 1999, the applicant submitted general comments and technical observations concerning the cited prior art. Given this situation, the applicant could not have been surprised by the Division's position and the discussion at the oral proceedings which essentially concerned the issue of inventive step of the claimed subject matter *vis-à-vis* the disclosure of document D1 and, at a later stage, the reasoning which formed the basis for refusing the

application. A procedural violation under Article 113(1) EPC as alleged by the appellant is, therefore, not discernable to the Board.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:

V. Commare

W. D. Weiß