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Case Number: T 1796/14 - 3.2.03

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Language of the proceedings: EN

Title of invention:
Method of manufacturing bulk metallic structures with submicron grain sizes and structures made with such method

Patent Proprietor:
H.C. STARCK, Inc.

Opponent:
Siemens Aktiengesellschaft

Headword:

Relevant legal provisions:
EPC Art. 56

Keyword:
Inventive step - non-obvious solution

Decisions cited:

Catchword:

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Case Number: T 1796/14 - 3.2.03

D E C I S I O N
of Technical Board of Appeal 3.2.03
of 4 May 2017

Appellant: (Opponent)	Siemens Aktiengesellschaft Wittelsbacherplatz 2 80333 München (DE)
Respondent: (Patent Proprietor)	H.C. STARCK, Inc. 45 Industrial Place Newton, Massachusetts 02461-1951 (US)
Representative:	Lux, Berthold Maiwald Patentanwalts GmbH Elisenhof Elisenstraße 3 80335 München (DE)
Decision under appeal:	**Decision of the Opposition Division of the European Patent Office posted on 4 July 2014 rejecting the opposition filed against European patent No. 2172292 pursuant to Article 101(2) EPC.**

Composition of the Board:

Chairman	G. Ashley
Members:	B. Miller
	M. Blasi

Summary of Facts and Submissions

I. European patent No. 2 172 292 relates to a method of manufacturing bulk metallic structures by a cold spray process. The grant of the patent was opposed on the basis of the grounds of opposition pursuant to Article 100(a) (novelty and inventive step) and 100(b) EPC.

II. The opposition against the patent was rejected by the opposition division. The appellant (opponent) filed an appeal against this decision in due time and form. The appeal was only directed against the opposition division's decision on inventive step. The other grounds for opposition were not further pursued.

III. The appellant requested that the decision under appeal be set aside and the patent be revoked.

IV. The respondent requested that the appeal be dismissed. Alternatively it was requested that the patent be maintained in amended form on the basis of one of the sets of claims filed as auxiliary requests 1 to 3 together with the response to the statement of grounds of appeal.

V. Claim 1 of the main request corresponds to granted claim 1 and reads:

"A process for producing three dimensionally large metallic structures comprised of submicron grain sizes, the process comprising:
using a cold spray system, accelerating a metal powder having a grain size larger than 5 microns with a heated

gas, thereby forming a supersonic metal powder jet; and
directing the supersonic metal powder jet against a substrate,
the powder adhering to the substrate and to itself to form a dense cohesive deposit having a submicron grain structure and a thickness of 1 cm or more."

Claims 2 to 11 of the main request relate to preferred embodiments of the process according to claim 1.

VI. State of the art

The following documents were cited in the appeal proceedings:

Q1: E. Calla et al., "Effect of Deposition Conditions on the Properties and Annealing Behavior of Cold-Sprayed Copper", J. Thermal Spray Technology 15(2), 2006, pages 255 to 262
Q2: T. Stoltenhoff et al., "Optimization of the Cold Spray Process", Proc. International Thermal Spray Conference, 28 to 30 May 2001, Singapore, 2001, pages 409 to 416
Q3: J. Pattison et al., "Cold gas dynamic manufacturing: A non-thermal approach to freeform fabrication", Int. J. Machine Tools & Manufacture, 47, 2007, pages 627 to 634
Q4: T. Schmidt et al., "Development of a generalized parameter window for cold spray deposition", Acta Materialia 54, 2006, pages 729 to 742
Q5: Entry from Wikipedia concerning "Lavaldüse"
Q6: Excerpt from Brockhaus Naturwissenschaft und Technik, 2003, pages 1323 to 1324
Q7: W. Bergmann, Werkstofftechnik, Teil 1, 1989, pages 264, 265 and 284 to 287

Q8: C. Borchers et al., "Microstructural bonding features of cold sprayed face centered cubic metals", Journal of Applied Physics, 2004, pages 4288 to 4292

Q9: M. Bohn et al., "Localization Microscopy Reveals Expression-Dependent Parameters of Chromatin Nanostructure", Biophys J., 2010, 99, pages 1358 to 1367

Q10: C. Cremer, "Lichtmikroskopie unterhalb des Abbe-Limits", Phys. Unserer Zeit, 2011, 42, pages 21 to 29.

Q11: R. W. Hertzberg, "Deformation and fracture mechanics of engineering materials", 3rd edition, John Wiley & Sons, New York, 1989, page 392.

VII. With the summons to oral proceedings, the Board sent a communication pursuant to Articles 15(1) and 17(2) of the Rules of Procedure of the Boards of Appeal (RPBA) indicating to the parties its preliminary opinion of the case.

VIII. Oral proceedings took place on 4 May 2017.

IX. The appellant's arguments can be summarised as follows.

The claimed process lacked an inventive step.
Document Q1 represented the closest prior art, since it proposed a cold spray process for providing a copper coating which could be removed from the substrate resulting in a three-dimensionally large structure. The critical speed for depositing copper was clearly above 570 m/s, i.e. supersonic, as evidenced by Q2, Q4 and Q8. Copper deposits formed by cold spraying according to Q1 had a submicron grain structure as confirmed by Q8. The features "deposit having a submicron grain

structure" and "forming a supersonic metal powder jet" of claim 1 therefore were inherently disclosed by Q1. The claimed process thus differed from that of Q1 only in terms of the defined thickness of deposit.
The mechanical integrity of the samples tested in Q1 would have motivated the skilled person to increase further the thickness of the samples to prepare samples with a thickness of 1 cm or more.
Furthermore, it was known in the art as evidenced by Q2 and Q3 that three-dimensionally large structures with a thickness of more than 1 cm could be produced by cold spray techniques.

The subject-matter of the claims as granted was therefore obvious in view of Q1 as the closest prior art, in particular when considering the teaching of Q2 or Q3.

X. The respondent's arguments can be summarised as follows.

Document Q1 proposed a process for providing a coating and not a three-dimensional structure.

The subject-matter of claim 1 of the contested patent differed not only in that the deposit formed by the process had a thickness of 1 cm or more but also in that the deposit had a submicron grain structure and in that a supersonic metal powder jet was used.

No hint could be found in any of the prior art documents that three-dimensional, large metal structures having a thickness of 1 cm or more and a submicron grain structure could be produced using a supersonic metal powder jet.

The subject-matter of the claims as granted therefore fulfilled the requirements of Article 56 EPC.

Reasons for the Decision

1. **Inventive step** (Article 100(a) and Article 56 EPC)

1.1 Disclosure of document Q1

Inventive step has been challenged by the appellant on the basis of document Q1 as the closest prior art.

Q1 discloses a cold gas dynamic spray deposition process using an in-house-designed de Laval nozzle, and helium as the driving gas operating at 298 and 523 K. Copper powder having a grain size between 5 and 10 µm is used as feedstock (page 256, sections 2.2 and 3.1).

A coating sample having a thickness of 2 mm is manufactured and its mechanical properties are evaluated (section 2.4 of Q1). Moreover, it is also indicated in section 2.2 of Q1 that a deposit having a thickness of 5 mm was obtained.

1.2 Distinguishing feature

The subject-matter of claim 1 of the patent as granted differs from the disclosure in Q1 at least in that the deposited structure has a thickness of 1 cm or more.

The disclosure of the following features was contested by the parties:

i) forming a supersonic metal powder jet; and

ii) a deposit having a submicron grain structure.

1.2.1 "forming a supersonic metal powder jet"

(a) Interpretation of the expression

The Board observes that the expression "forming a supersonic metal powder jet" in claim 1 does not clearly define whether the carrier gas or the carrier gas together with the metal particles have to have supersonic speed.

In the present case it is said in paragraph [0012] of the contested patent "that certain metal powders of conventional grain size, substantially 5-10 microns and even larger, when projected at supersonic velocity, at relatively low temperature and deposited on a substrate form a dense solid having a submicron grain structure”.

Thus the description of the contested patent renders it clear that the metal powder itself has to have supersonic speed.

(b) Teaching of Q1

Q1 itself does not disclose the speed of the particles during the spraying.

Q4 (left column, 3rd sentence) confirms the general knowledge of the skilled person that the impact speed of particles in cold spraying lies between 200 - 1200 m/s.

However, it is also general knowledge that, depending on the powder material, a critical minimum speed is required to achieve a successful deposition. In case of a copper powder, which is the powder used according to the cold spraying method described in Q1, the particles need to have a critical speed of at least about 570 m/s, as evidenced by Q2 (page 412, second last sentence), Q4 (Figure 7) and Q8 (page 4291, left column, line 5), in order to achieve the required deposition.

The Board therefore can accept that the skilled reader of Q1 would implicitly understand that the copper particles must be accelerated at least to the critical speed in order to achieve the intended deposition. Therefore it can be concluded that in the method proposed by Q1 the copper particles have a speed faster than sound in air (about 340 m/s).

1.2.2 "a deposit having a submicron grain structure"

Q1 does not explicitly disclose that the deposited metal coating has a submicron grain structure.

The appellant argued that the grain size was implicitly derivable from Q1 by considering

a) the grain size after annealing,
b) the images of figure 3,
c) the reported high dislocation and mechanical properties and
d) the reference to Q8.

concerning point a)

A specific disclosure concerning the grain size can be found on page 261, line 14 of the left column where it is stated that the grain size after annealing at 773 K is between 1 and 5 µm when the deposit is formed by spraying with helium at 523K.

An annealing process has an impact on the grain size, e.g. due to possible recrystallisation processes and grain growth. Although it is likely that the annealed grain size is greater than that of the material as deposited, it cannot be said with any certainty that the grain size obtained during the deposition process was submicron.

concerning point b)

Figure 3 shows optical micrographs of the as-sprayed deposited material wherein the grain size cannot be clearly determined.

Contrary to the argument of the appellant, the Board cannot conclude that the absence of visible grains inherently leads to the conclusion that the grain size is submicron, since the fact that grains are not visible in figure 3 of Q1 could be due to one of several reasons and could for example result from the fact that the chosen resolution of the microscope used for taking the picture was too low.

In this context it is not considered to be relevant whether or not optical microscopy is a technique in principle suitable for making submicron grains visible. There is no clear indication in Q1 under which conditions the pictures of figure 3 were taken and what the reasons are for not seeing the grains.

Speculation based on the fact that the grain size is not shown in figure 3 of Q1 is insufficient to establish the grain size with any certainty.

concerning point c)

A high dislocation density in the copper coating might explain the tensile strength measurements reported in chapter 4.1 and figure 8 of Q1. However, a direct link between the measurements reported in Q1 and the actual grain size has not been established. Therefore in principle the same argumentation as with respect to the conclusions drawn from figure 3 applies. A certain likelihood is not sufficient to establish that a feature is inherently disclosed.

concerning point d)

The appellant further argued that a submicron grain structure was inevitably obtained by the deposition method of Q1, as evidenced by Q8 which shows in figure 4 a TEM micrograph of a cold spray copper coating having a submciron grain size.

Document Q8 is referred to in Q1 as reference 8. Contrary to the argument of the appellant, the disclosure of Q8 is not incorporated into Q1, in particular as it is said in section 4.1 (page 260, right hand column, first complete sentence):

"However, the current work indicates that the microstructure is sensitive to spraying conditions, and thus it is difficult to compare the work of Borchers et al. (Ref 6, 8, 15) with the current work."

Q1 therefore explicitly states that the relevant teaching of Q8 concerning the microstructure of the deposited metal cannot be compared with that of Q1.

Hence the submicron grain structure shown in figure 4 of Q8 is not necessarily achieved when following the teaching of Q1.

Summary concerning points a) to d)

In view of the considerations provided above, the Board concludes that Q1 does not directly and unambiguously discloses a process leading to a metal deposit having a submicron grain structure.

1.2.3 The process of claim 1 differs from the method of Q1 at least in that a deposit having a submicron grain structure and a thickness of 1 cm or more is formed.

1.3 *Objective technical problem*

As illustrated in paragraph [0014] of the contested patent, the submicron grain size improves interparticle bond strength, eliminates work hardening and improves ductility.

Starting from the disclosure of Q1, the technical problem to be solved can be formulated as providing a process which results in three-dimensionally larger metallic structures with improved mechanical properties.

1.4 *Non-obviousness of the solution of the problem*

1.4.1 Q1 is directed to a process for depositing oxide-free coatings. The coating produced by Q1 was thick enough such that it could be carefully removed and the mechanical properties determined (section 2.4 of Q1).

1.4.2 Starting from this disclosure, the appellant argued that no teaching could be found that would prevent the skilled person from simply producing thicker coatings by using more passes of the spray gun over the substrate.

However, the absence of an indication not to increase the thickness is no indication to a skilled person that the thickness of the coating obtained by Q1 can be substantially increased by at least 100 % from 5 mm to at least 1 cm, in particular since Q1 aims at providing a coating (see abstract) and not necessarily at producing large, three-dimensional structures.

1.4.3 The appellant further argued that Q1 teaches that good mechanical properties are achieved, such as the hardness of the deposits before and after annealing at certain temperatures (section 3.4 of Q1) and the reported high dislocation density (section 4.1 of Q1), and that these are a clear pointer that thicker structures could be produced.

However, the Board observes that in order to arrive at the subject-matter of claim 1 it is not only necessary to achieve a thicker structure but also to achieve a thicker structure having a submicron grain structure. A link between the presence of a submicron grain size in the deposited coating and its mechanical properties as indicated in paragraph [0014] of the contested patent is not derivable from Q1, since the grain structure of the deposit as such is not addressed by Q1.

Therefore even if the skilled person had concluded in view of the mechanical properties reported by Q1 that a thicker structure could be produced, he had no reasonable expectation that a structure having a submicron grain size leading to the beneficial properties reported in the contested patent would be created in a thicker deposit.

1.4.4 Hence, the Board concludes that Q1 on its own does not provide any hint for the skilled person that the process can be used to obtain three-dimensionally large metallic structures with a thickness of more than 1 cm while achieving a submicron grain size leading to improved mechanical properties.

1.4.5 The appellant further argued that claim 1 was obvious when starting from Q1 and taking into account Q3 in addition.

It is known from Q3 that large structures such as shown in figures 5, 6 or 7 can be obtained by using cold gas dynamic manufacturing. However, the process of Q3 is described as a new cold gas dynamic manufacturing technique for freeform fabrication which combines additive and subtractive techniques to enable the production of complex geometries (abstract).
Given the differences between the techniques of Q1 and Q3 it is doubtful that the skilled person would turn to Q3 for the purpose of developing the process of Q1.

However, even if the skilled person consulted Q3, he gets no incentive to modify the process described by Q1 in order to arrive at claim 1.

Concerning the cold spray (CS) process Q3 states on page 628, left hand column, second paragraph by reference to further articles (references 12 and 13 of Q3) that "the CS process has been frequently used to deposit temperature-sensitive materials such as nano-crystalline and amorphous materials ...".

However this general statement neither teaches that the novel method described in Q3 provides structures with a submicron grain size nor suggests, that a submicron grain size should be obtained in a cold spray process to achieve the advantageous properties described in paragraph [0014] of the contested patent.

The general statement in Q3 thus provides no incentive to modify the process described in Q1 by providing a thicker deposit and by adjusting the process to obtain a submicron grain size to produce three-dimensionally larger metallic structures with improved mechanical properties.

The Board therefore concludes that the subject-matter of claim 1 of the contested patent is not obvious when starting from Q1 and taking into account the teaching of Q3.

1.4.6 The appellant further argued that claim 1 was obvious when starting from Q1 and taking into account Q2.

Q2 suggests that structures having a thickness of several centimetres can be obtained by a cold spray process (page 415, chapter "summary and conclusions", first paragraph). However, Q2 does not suggest to provide a deposit having a submicron grain structure by using a supersonic metal powder jet.

Therefore the Board comes to the conclusion that the subject-matter of claim 1 as granted is not obvious when starting from Q1 as the closest prior art and taking into account Q2.

2. In summary, none of the inventive step objections starting from Q1 raised by the appellant prejudice the maintenance of the patent as granted under Article 100(a) and Article 56 EPC.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

C. Spira G. Ashley

Decision electronically authenticated