

DATE-REINFORCEMENT OF TITANIUM
COMPOSITES WITH BORIDES (*TITLE OF PhD THESIS*)

BY
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RESPONSE TO THE THIRD REVIEWER'S COMMENTS-(S2010/1375)

The reviewer listed some corrections expected that the candidate should attend to. Below are the corrections and the corresponding response of the candidate.

- Examiner (S2010/1375):** Page 5, line19: this can be ò achieveö not-achieved

Response of the candidate: In line 19 of page 5, òachieveö has been changed to òachievedö.
- Examiner (S2010/1375):** Page 17, line 8: that òresultedö from- results

Response of the candidate: In line 8 of page 17 òresultedö has been changed to òresultsö
- Examiner (S2010/1375):** Page 17, line 12: recent research now focuses-
change to recent research focuses.

Response of the candidate: On page 17, line 12: òrecent research now
focusesö has been changed to òrecent research focusesö
- Examiner (S2010/1375):** Page 21, line 11: sentence starting with òthe present
study thereforeö requires paraphrasing as it does not make any sense.

Response of the candidate: On page 21, line 11: The sentence has been
reconstructed to now read òBased on the convinced reports on the wide
acceptability of this method in the production of titanium alloy composites
(because of its economic reliability and cost effectiveness), the present study
considered the method has an appropriate, a more suitable and affordable
production route for TMCö
- Examiner (S2010/1375):** Page 29, line 3: is it equ. 2.3 or equation 2.3? The
candidate needs to be consistent on the format in the thesis.

Response of the candidate: On page 29, line 11&12: òequ.ö represents
òequationö. For consistency, òequationö has been changed to òequ.ö

5): Page 46, Fig 2.11: values on both vertical axes are not clear

Response of the candidate: Page 46, Fig 2.11: The picture has been adjusted and the values on both vertical axes are now clearer with left vertical axis represents Weight (%) and right vertical axis represents Temperature difference ($^{\circ}\text{C}/\text{mg}$).

7. **Examiner (S2010/1375):** Page 50, line 18: sentence starting with "final no reported" requires paraphrasing as it does not make any sense.

Response of the candidate: The correction is on Page 50, line 18: The sentence now reads thus "The extensive review carried out in the present study shows that no research work has been reported on the use of another source of titanium like titanium hydride powders as metal matrix based and available review indicates the possibility of using titanium hydride as an alternative source of titanium matrix in the TMCs fabrication."

8. **Examiner (S2010/1375):** Page 52, line 8: fig 3.1 should read Fig 3.1.

Response of the candidate: The correction is on Page 52, line 8: fig 3.1 has been changed to Fig. 3.1.

9. **Examiner (S2010/1375):** Page 54, Figure 3.2: the candidate needs to identify the components of the equipment by labeling them.

Response of the candidate: The correction is on Page 54, Figure 3.2: To avoid complexity, the picture of the equipment, maker, model and year were presented.

10. **Examiner (S2010/1375):** Page 55, Figure 3.3: the candidate needs to identify the components of the equipment by labeling them.

Response of the candidate: The correction is on Page 55, Figure 3.3: To avoid complexity, the picture of the equipment, maker, model and year were presented.

11. **Examiner (S2010/1375):** Page 56, Figure 3.4: the candidate needs to identify the components of the equipment by labeling them.

- idate:* The correction is on Page 56, Figure 3.4: To avoid complexity, the picture of the equipment, maker, model and year were presented.
12. **Examiner (S2010/1375):** Page 57, Figure 3.5: the candidate needs to identify the components of the equipment by labeling them.
Response of the candidate: The correction is on Page 57, Figure 3.5: To avoid complexity, the picture of the equipment, maker, model and year were presented.
13. **Examiner (S2010/1375):** Page 58, Figure 3.6: the candidate needs to identify the components of the equipment by labeling them.
Response of the candidate: The correction is on Page 58, Figure 3.6: To avoid complexity, the picture of the equipment, maker, model and year were presented.
14. **Examiner (S2010/1375):** Page 59, Figure 3.7: the candidate needs to identify the components of the equipment by labeling them.
Response of the candidate: The correction is on Page 59, Figure 3.7: To avoid complexity, the picture of the equipment, maker, model and year were presented.
15. **Examiner (S2010/1375):** Page 65, line 2: öis giving in equation 3.1ö should read öis given in equation 3.1ö
Response of the candidate: The correction is on Page 65, line 2: öis giving in equation 3.1ö has been changed to öis given in equation 3.1ö
16. **Examiner (S2010/1375):** Page 68, line 4: is the 10% not supposed to be 20% in the cooling regime used?
Response of the candidate: The correction is on Page 68, line 4: 10% has been changed to 20% and the statement now read as öwhile 20HPTMC1100 represent a 20vol. % reinforced particulate TMC hot-pressed sintered at 1100°Cö
17. **Examiner (S2010/1375):** Page 69, line 17: öequations 3.5 & 3.6 was usedö should read öequations 3.5 & 3.6 was usedö

- late:* The correction is on Page 69, line 17: "equation 3.5 & 3.6 was used" has been changed to "equations 3.5 and 3.6 were used"
18. **Examiner (S2010/1375):** Page 72, line 7: "SEM/EDX analyses results" should read "SEM/EDX analysis results"
Response of the candidate: The correction is on Page 72, line 7: "SEM/EDX analyses results" has been changed to "SEM/EDX analysis results"
19. **Examiner (S2010/1375):** Page 75: Dehydrogenation of TiH_2 : the candidate states that the dehydrogenation of TiH_2 powder at 680°C led to complete conversion to Ti phase without stating that this was a function of residence time. Table 4.1 indicates that complete conversion was only achieved at dwelling time of 120 min-can candidate revisit that comment.
Response of the candidate: The correction is on Page 75: The statement has been reorganized and it now read as "The loose TiH_2 powder was dehydrogenated at 680°C after the dwelling time of 120mins, which shows the significance of dwelling time in achieving pure Ti phase (Table 4.1)"
20. **Examiner (S2010/1375):** Page 76, line 14: " TiH_2 based materials start to densified at" - densify
Response of the candidate: The correction is on Page 76, line 14: " TiH_2 based materials start to densified at" has been changed to " TiH_2 based materials start to densify at" (densified changed to densify).
21. **Examiner (S2010/1375):** Page 81, line 4: full stop after "results" is missing
Response of the candidate: The correction is on Page 81, line 4: full stop has been placed after "results"
22. **Examiner (S2010/1375):** Page 82 not 71, line 9: place a full stop between A1 and Table 4.3.
Response of the candidate: The correction is now on Page 82, line 9: full stop has been placed after A1.
23. **Examiner (S2010/1375):** Page 83, line 11: Fig (4.7b) is not a XRD pattern-please rectify and indicate which figure is being referred to.

Candidate: The correction is on Page 83, line 11: Fig.

(4.7b) is the SEM/EDX micrograph of the mixed powder used to fabricate 20vol% TMCs. The corrected statement now read as ~~the~~ the same observation was made in the XRD pattern (Fig. 4.6b) and SEM/EDX (Fig.4.7b) for mixed powder prepared for the fabrication of 20 vol. % reinforced titanium matrix composite (TMC)~~ø~~

24. **Examiner (S2010/1375):** Page 86, Fig 4.7 (c, d & e): these figures are not clear at all. The candidate should present higher quality scans.

Response of the candidate: The correction is on Page 86: Better quality scans were reproduced and readjusted for the SEM micrographs in Fig. 4.7 (a-e). All the figures are now presented in colour for with higher quality.

25. **Examiner (S2010/1375):** Page 94, line 24: ~~the~~ the reduction in number and needle shaped TiB~~ø~~ should read ~~ø~~ the reduction in number of needle shaped TiB~~ø~~

Response of the candidate: The correction is on Page 94, line 24: ~~the~~ the reduction in number and needle shaped TiB~~ø~~ has been changed to ~~ø~~ the reduction in number of needle shaped TiB~~ø~~

26. **Examiner (S2010/1375):** Page 96, Fig. 4.13 SEM (a) and (b): the phases that are pointed to by TiB arrows do not show clearly any spiky TiB phases as the candidate suggest

Response of the candidate: The correction is on Page 96, Fig. 4.13 SEM (a) and (b): The TiB arrows were adjusted to point at the spongy structures that denotes TiB. The spongy structures (at higher magnification) shows aggregate of needle shaped TiB.

27. **Examiner (S2010/1375):** Page 101, line 5: ~~ø~~sinter~~ø~~ should read ~~ø~~sinter~~ø~~

Response of the candidate: The correction is on Page 101, line 5: ~~ø~~sinter~~ø~~ has been changed to ~~ø~~sinter~~ø~~

28. **Examiner (S2010/1375):** Page 102, line 21: ~~ø~~Analyses of~~ø~~ should read ~~ø~~Analysis of~~ø~~

- late:* The correction is on Page 102, line 21: ~~Analyses~~ of ~~ø~~ has been changed to ~~Analysis of~~ ~~ø~~
29. **Examiner (S2010/1375):** Page 110, line 9: ~~produced~~ should read ~~produce~~
- Response of the candidate:* The correction is on Page 110, line 9: ~~produced~~ has been changed to ~~produce~~
30. **Examiner (S2010/1375):** Page 152, line 7: which might thus reduce-which might reduce
- Response of the candidate:* No such statement was found on the specified page and line.
31. **Examiner (S2010/1375):** Page 106, 107, 108, 109: SEMs in Fig. 4.21; Fig. 4.23; Fig. 4.25; Fig. 4.27; need improvement, the phases indicated as TiC and TiB are not visible as the candidate would like the reader to believe.
- Response of the candidate:* The correction is on Page 106, 107, 108, and 109: SEMs in Fig. 4.21; Fig. 4.23; Fig. 4.25; Fig. 4.27 shows clearly the type of TiC and TiB synthesized in those regimes (which agreed with relevant literature), presentation of the pictures in black and white makes it look unclear. All the pictures were reproduced, readjusted and presented in colour, which now enhanced the quality. Morphological transformation of the structures was extensively explained on page 105 and 106.
32. **Examiner (S2010/1375):** Page 113, line 4: T-B₆O should read Ti-B₆O
- Response of the candidate:* The correction is on Page 113, line 4: ~~T-B₆O~~ has been changed to ~~Ti-B₆O~~
33. **Examiner (S2010/1375):** Page 113, Table 4.11: The trend for the fracture toughness is not explained. Can the candidate offer an explanation of the behavior of the measured fracture toughness in relation to the microstructure of product samples for the different experimental conditions.
- Response of the candidate:* The correction is now on Page 149: As shown in table 4.11, the fracture toughness of hot-pressed Ti-B₆O composites changes with sintering temperature, as well as the composition of the reinforcements

There is no consistency in the values obtained. For instance, sample **HP8Ti2B₆O1000** has fracture toughness of 10.6MPa.m^{1/2} while that of sample **HP8Ti2B₆O1100** is 7.3MPa.m^{1/2}.

In comparison, hot-pressed Ti-B₆O composites shows higher fracture toughness compared to pressureless and hot-pressed Ti-B₄C composites produced. For instance, sample **HP8Ti2B₆O1300** show fracture toughness of 10.8MPa.m^{1/2} compared to 5.3MPa.m^{1/2} shown by sample **40HPTMC1400**. The observed difference could be attributed to difference in composition and contents of reinforcements, as well as reinforcement's morphology and the quantity that occur during the heat treatment (Dubey and Soboyejo, 1997). This claim is substantiated with the observation from the XRD (Fig. 4.31, page 116) and SEM (Fig. 4.32, page 117) analysis of Ti-B₆O composites that shows the presence of Ti₂O, TiB, TiB₂ and unreacted B₆O phases against Ti, TiC and TiB phases detected in Ti-B₄C composites.

34. **Examiner (S2010/1375):** Page 114- the candidate observe that at sintering temperatures of 1200°C the hardness decreases slightly, can he give a suggestion as to why is so.

Response of the candidate: The correction is on page 114. The slight decrease in hardness observed at temperature above 1200°C could be as a result of some experimental errors resulting from the indentation points used in calculating the hardness.

35. **Examiner (S2010/1375):** Page 119, line 9: "Compaction" should read "Compaction"

Response of the candidate: The correction is on Page 119, line 9: "Compaction" has been changed to "compaction"

36. **Examiner (S2010/1375):** Page 121, line 6: "Analyses" should read "Analysis"

- Candidate:** The correction is on Page 121, line 6:
"Analyses" has been changed to "Analysis"
37. **Examiner (S2010/1375):** Page 123, line 27: "and bonding of it particles" should read "and bonding of its particles"
Response of the candidate: The correction is on Page 123, line 27: "and bonding of it particles" has been changed to "and bonding of its particles"
38. **Examiner (S2010/1375):** Page 126, line 1: "The study undertake here" should read "The study undertaken here"
Response of the candidate: The correction is on Page 126, line 1: "The study undertake here" has been changed to "The study undertaken here"
39. **Examiner (S2010/1375):** Page 131, line 3: The observations made do not appear to agree with the curve according to Alman and Hawk as the candidate suggests. That statement needs further clarification.
Response of the candidate: The correction is on Page 131, line 1-3:
The statement now read "These results shows that the relative density of Ti matrix composite produced decreases with increasing volume fraction of inclusion. This observation, as shown in figure 5.11 agreed well with findings of Saito, *et al.*, (1998) and Alman and Hawk, (1999)"
40. **Examiner (S2010/1375):** Page 133, line 2: "the microstructures analyses" should read "the micro structural analysis"
Response of the candidate: The correction is on Page 133, line 2: "the microstructures analyses" has been changed to "the micro structural analysis"