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In-situ particulate-reinforcement of titanium matrix composites with borides

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

BY

ABDULFATAI JIMOH

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.

1. **Examiner (S2010/1375):** Page 5, line19: this can be ñ achieveò not-achieved
   Response of the candidate: In line 19 of page 5, ñachieveò has been changed to ñachievedò.
2. **Examiner (S2010/1375):** Page 17, line 8: that ñresultedò from- results
   Response of the candidate: In line 8 of page 17 ñresultedò has been changed to ñresultsò
3. **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: ñrecent research now focusesò has been changed to ñrecent research focusesò
4. **Examiner (S2010/1375):** Page 21, line 11: sentence starting with ñthe present study thereforeò 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 ñBased 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 TMCò
5. **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: ñequ.ò represents ñequationò. For consistency, ñequationò has been changed to ñequ.ò

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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 (^{o}C/mg).

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

   Response of the candidate: The correction is on Page 50, line 18: The sentence now read thus ñThe 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.

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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: ñis giving in equation 3.1ò should read ñis given in equation 3.1ò

    Response of the candidate: The correction is on Page 65, line 2: ñis giving in equation 3.1ò has been changed to ñis given in equation 3.1ò

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 ñwhile 20**HP**TMC**1100** represent a 20vol. % reinforced particulate TMC hot-pressed sintered at 1100^{0}Cñ

17. **Examiner (S2010/1375):** Page 69, line 17: ñequations 3.5 & 3.6 was usedò should read ñequations 3.5 & 3.6 was usedò

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late: The correction is on Page 69, line 17: ñequation 3.5 & 3.6 was usedò has been changed to ñequations 3.5 and 3.6 were usedò

18. **Examiner (S2010/1375):** Page 72, line 7: ñSEM/EDX analyses resultsò should read ñSEM/EDX analysis resultsò

    Response of the candidate: The correction is on Page 72, line 7: ñSEM/EDX analyses resultsò has been changed to ñSEM/EDX analysis resultsò

19. **Examiner (S2010/1375):** Page 75: Dehydrogenation of TiH_2: the candidate states that the dehydrogenation of TiH_2 powder at 680^oC 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 ñThe loose TiH_2 powder was dehydrogenated at 680^oC after the dwelling time of 120mins, which shows the significance of dwelling time in achieving pure Ti phase (Table 4.1)ò

20. **Examiner (S2010/1375):** Page 76, line 14: ñTiH_2 based materials start to densified at ò - densify

    Response of the candidate: The correction is on Page 76, line 14: ñTiH_2 based materials start to densified atò has been changed to ñTiH_2 based materials start to densify atò (densified changed to densify).

21. **Examiner (S2010/1375):** Page 81, line 4: full stop after ñresultsò is missing

    Response of the candidate: The correction is on Page 81, line 4: full stop has been placed after ñresultsò

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.

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lidate: 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 ôthe 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)öö

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: ôöthe reduction in number and needle shaped TiBöö should read ôö the reduction in number of needle shaped TiBöö

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

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: ôösintereöö should read ôösinteröö

    Response of the candidate: The correction is on Page 101, line 5: ôösintereöö has been changed to ôösinteröö

28. **Examiner (S2010/1375):** Page 102, line 21: ôöAnalyses oföö should read ôöAnalysis oföö

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late: The correction is on Page 102, line21: ôôAnalyses ofôô has been changed to ôôAnalysis ofôô

29. **Examiner (S2010/1375):** Page 110, line 9: ôôproducedôô should read ôôproduceôô

    Response of the candidate: The correction is on Page 110, line 9: ôôproducedôô has been changed to ôôproduceôô

30. **Examiner (S2010/1375):** Page 152, line 7: which might thus reduce-which might reduceô

    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-B_6O should read Ti-B_6O

    Response of the candidate: The correction is on Page 113, line 4: ñT-B_6Oò has been changed to ñTi-B_6Oò

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-B_6O composites changes with sintering temperature, as well as the composition of the reinforcements

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is no consistency in the values obtained. For instance, sample **HP**8Ti2B_6O**1000** has fracture toughness of 10.6MPa.$m^{1/2}$ while that of sample **HP**8Ti2B_6O**1100** is 7.3MPa.$m^{1/2}$.

In comparison, hot-pressed Ti-B_6O composites shows higher fracture toughness compared to pressureless and hot-pressed Ti-B_4C composites produced. For instance, sample **HP**8Ti2B_6O**1300** show fracture toughness of 10.8MPa.$m^{1/2}$ compared to 5.3MPa.$m^{1/2}$ shown by sample 40**HP**TMC**1400**. The observed difference could be attributed to difference in composition and contents of reinforcements, as well as reinforcementôs 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-B_6O composites that shows the presence of Ti_2O, TiB, TiB_2 and unreacted B_6O 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^oC 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^oC 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: ñCCompactionò should read ñCompactionò

    Response of the candidate: The correction is on Page 119, line 9: ñCCompactionò has been changed to ñcompactionò

36. **Examiner (S2010/1375):** Page 121, line 6: ñAnalysesò should read ñAnalysisò

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ndidate: The correction is on Page 121, line 6: ñAnalysesò has been changed to ñAnalysisò

37. **Examiner (S2010/1375):** Page 123, line 27: ñand bonding of it particlesò should read ñand bonding of its particlesò

    Response of the candidate: The correction is on Page 123, line 27: ñand bonding of it particlesò has been changed to ñand bonding of its particlesò

38. **Examiner (S2010/1375):** Page 126, line 1: ñThe study undertake hereò should read ñThe study undertaken hereò

    Response of the candidate: The correction is on Page 126, line 1: ñThe study undertake hereò has been changed to ñThe study undertaken hereò

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 ñThese 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)ò

40. **Examiner (S2010/1375):** Page 133, line 2: ñthe microstructures analysesò should read ñthe micro structural analysisò

    Response of the candidate: The correction is on Page 133, line 2: ñthe microstructures analysesò has been changed to ñthe micro structural analysisò