CHAPTER – 6

Summary

and

Suggestions for Future Work

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SUMMARY AND SUGGESTIONS FOR FUTURE WORK

The significant findings of the present work are summarized in this chapter. The structural transformation and thermal stability of nanocomposite powders, and microstructural features and mechanical properties of the sintered composites were critically studied. The key findings and observations with regards to the nanocomposite powder and bulk composite were discussed in the respective chapters. However, the overall conclusions and comments as well as suggestion for future work are briefly summarized as follows:

6.1 Summary

In the present work, systematic efforts were made towards the synthesis of Al-Cu-Fe quasicrystalline matrix with soft Sn reinforcement, and AA 6082 Al matrix composite reinforced with Al-Cu-Fe quasicrystalline and non-equiatomic AlSiCrMnFeNiCu HEA through mechanical milling. The mechanical milling of IQC-Sn, Al-IQC and Al-HEA results in the nanostructuring of these composites along with the homogenous distribution of reinforcement in the matrix.

6.1.1 Al-Cu-Fe quasicrystalline matrix composite reinforced with Sn

The IQC alloy used in the present work was synthesized by vacuum induction melting. The as-cast IQC alloy was subjected to a suitable annealing treatment at 800 °C for synthesizing stable icosahedral phase. However, even after annealing a minor fraction of $Al_{13}Fe_4$ and B2-type Al(Cu, Fe) was found to be remaining in the IQC alloy. The grain refinement, sequence of structural transformation, indentation behaviour was found to be varying as a function of milling duration and volume fraction of Sn in the IQC-Sn nanocomposite powders. It was observed that the Al-Cu-Fe IQC alloy reinforced with 10 vol% of Sn was found to have a prudential formation of nanocrystalline $Al_{13}Fe_4$ phase after 30 h of mechanical milling. However, nanoquasicrystalline IQC and nanocrystalline B2-

type Al (Cu, Fe) has evolved as a minor phase after 30 h. Further, the Al-Cu-Fe IQC alloy was able to retain its face-centred ordering until 20 h of mechanical milling and becomes disordered after that as was evident with the disappearance of (311111) reflection of IQC alloy. In the case of Al-Cu-Fe IQC reinforced with 30 vol% of Sn, the sequence of structural transformation during mechanical milling is different than that with 10 vol% of Sn. The Al-Cu-Fe was able to retain its face-centred ordering up to 30 h of milling along with the prudential formation of B2-type Al(Cu, Fe) phase due to an increase in the volume fraction of Sn. On the contrary to IQC-10Sn, the nanocrystalline $Al_{13}Fe_4$ have minor phase fraction in IQC-30Sn. The thermal stability of these IQC-Sn nanocomposites were established through DSC and ex-situ XRD. Although, the phases formed at 800 °C (1073 K) showed its dependence on the volume fraction of Sn reinforcement, however, only IQC and crystalline phases of $Al_{13}F_4$ and B2-type Al (Cu, Fe) was evident. Further, the ex-situ XRD of IQC-Sn nanocomposite powder confirmed that at higher temperatures, Sn reinforcement was able to retain its identity even after annealing for 10 h. The milling duration and volume fraction of Sn reinforcement in IQC-Sn nanocomposite influence its indentation behaviour too. The hardness of these nanocomposites was found to be varying in between ~4 to 7 GPa.

The detailed structural and thermal studies, and indentation behaviour of Al-Cu-Fe IQC reinforced with Sn paved the path for the bulk composite through spark plasma sintering (SPS), hot-pressing (HP) and pressure-less sintering. The SPS of IQC-Sn composite has resulted in a bulk compact with a relative density of 100%. The phase evolved after SPS of IQC-Sn composite confirmed with dependence on the volume fraction of the Sn reinforcement as was the case for IQC-Sn nanocomposite milled for 40h. However, the SPSed composite the IQC phase was no longer appearing as a minor phase as it was in the IQC-Sn nanocomposite milled for 40 h. The SPSed IQC-Sn composite showed considerable enhancement in the compressive yield strength and was found to be ~544 MPa, 880 MPa and 500 MPa for IQC-10Sn, 20Sn and 30Sn respectively. The highest increase ~75% was observed for Al-Cu-Fe IQC alloy reinforced with 20 vol% of Sn reinforcement. The bulk composite prepared by HP has also shown promising results. The hardness of the HPed IQC-10Sn composite was found ~9.7 GPa at a load of 50 g coupled with 22% enhancement in the fracture toughness. The increase in the fracture toughness of these composite was attributed to the inhibition of the indentation cracks by soft Sn phases homogeneously dispersed in the IQC matrix. These properties of the Al-Cu-Fe IQC alloys reinforced with Sn reinforcement make them a suitable material for their engineering application like the coating of bearings etc.

6.1.2 Al-based metal matrix composite reinforced with Al-Cu-Fe quasicrystals

The Al-Cu-Fe IQC alloy was used as a reinforcement in the AA 6082 Al matrix composite through mechanical milling and SPS. The systematic investigation has been carried out to understand the effect of milling duration and volume fraction of IQC reinforcement on the grain refinement and structural transformation of both matrix as well as reinforcement. The structural transformation during milling was established through XRD and TEM. It was observed that during milling of Al-IQC nanocomposite up to 30 vol% of IQC, no structural change of either the matrix or the reinforcement was evident. However, the case is not same for Al-IQC nanocomposite reinforced with 40 vol% of IQC. In Al-40IQC it was observed that the IQC phase partially transforms into λ-$Al_{13}Fe_4$ phase after 20h of MM. Although, even after partial structural transformation of IQC to λ-$Al_{13}Fe_4$ phase, the face-centred ordering of IQC reinforcement was still prevalent as evident from the (311111) reflection of IQC phase. The milling duration and volume fraction of the reinforcement was found to enhance the nanostructuring of the Al matrix. Further, the IQC reinforcement was found to be well embedded in the Al matrix during milling for 50 h.

Increasing the IQC fraction in Al-IQC nanocomposite has led to significant crystallite size refinement and an increase in the lattice strain. However, the improvement in the crystallite size and increase in lattice strain was not very appreciable for Al-40IQC in comparison to Al-30IQC nanocomposite powder.

These Al-IQC nanocomposites were SPSed in two different processing condition. The Al-40IQC nanocomposite sample was sintered at 550 ℃ (823 K) with a pressure of 50 MPa for 30 min. However, the Al-10, 20 and 30 IQC nanocomposite powder was SPSed at 300 ℃ (573 K) with high pressure of 500 MPa for 30 min. The literature has suggested the formation of ω-Al_7Cu_2Fe phase during hot pressing of Al-IQC above 623 K. Therefore, the high pressure and low-temperature sintering protocol was adopted for retention of the IQC phase in Al-IQC composite. The phase analysis of Al-40IQC SPSed composite has suggested the formation of ω-Al_7Cu_2Fe phase, Al_2Cu phase along with a minor fraction of pre-existing phases of IQC and $Al_{13}Fe_4$. However, the phase analysis of Al-IQC SPSed composite suggested the formation of a significant IQC phase along with little fraction corresponding to the ω-Al_7Cu_2Fe and Al_2Cu phase. The compressive yield strength for Al-40IQC was found to ~519 MPa with an appreciable failure strain of ~6.0%. The compressive yield strength of Al-30IQC processed at 300 ℃ (573 K) with a pressure of 500 MPa was found to be ~900 MPa with a failure strain of ~4.5%. The enormous increase in the strength of Al-IQC SPSed composite was attributed to the direct and indirect strengthening (solid solution strengthening).

6.1.3 Al-based metal matrix composite reinforced with AlSiCrMnFeNiCu HEA

In this work, an unconventional approach has been adopted for designing AMCs reinforced with non-equiatomic AlSiCrMnFeNiCu HEA. The AA 6082 Al matrix composite was reinforced with these non-equiatomic HEA processed through mechanical milling and pressure-less sintering. Although the major objective in this work was the synthesis and characterization of Al-HEA composite, however, it was equally important to study the non-equiatomic HEA. The non-equiatomic HEA used in the present investigation belongs to the class of HEA having Al as the principal alloying elements. The non-equiatomic AlSiCrMnFeNiCu HEA was prepared by vacuum induction melting. The as-cast HEA was consisting of two phases corresponding to a major B2-type and a minor Cr_5Si_3 (tetragonal crystal structure). The structural examination of these HEA through XRD and TEM confirmed the ordering of B2-type phase as evident from the (100) superlattice reflection. The phase formation and its stability were corroborated with several thermodynamic models and property diagram generated by Thermo-Calc software. The thermodynamic parameters such VEC suggested the formation of a BCC solid solution phase. However, the other parameter such as 'Ω' suggested the appearance of a solid solution in the present alloy system was inevitable. This contradiction in the thermodynamic parameters can be attributed to the high positive enthalpy of mixing among binary Cr-Cu, Cu-Mn, and Cu-Fe, and increased negative enthalpy of mixing among Mn-Si, Cr-Si, Fe-Si, and Ni-Si.

These non-equiatomic HEA having very high hardness was then used as a reinforcement in AMCs. The non-equiatomic HEA was able to retain its identity even after mechanical milling of Al-HEA nanocomposite for 50 h. These non-equiatomic HEA has significantly refined the Al-HEA nanocomposite, and the level of crystallite size refinement and increase in lattice strain was higher in comparison to the Al-IQC nanocomposite powders. During MM of Al-30HEA nanocomposite powder for 50 h, a minimum grain size of ~10-12 nm was achieved. The Al-HEA nanocomposite powder was found to be thermally stable up to to 650 ℃ (923 K) till the melting of the Al matrix. The in-situ XRD at high temperature up to 560 ℃ (833 K) confirmed the claim for thermal stability of Al-HEA nanocomposite powders. Although, a few minor exothermic fluctuation was observed in the DSC thermogram, however, it can be attributed to the dissolution of alloying elements from non-equiatomic HEA into the Al matrix. This claim was further affirmed by the diffused nature of the Al matrix and HEA particles reinforcement after pressureless sintering at 560 ℃ (833 K). The SEM-EDS mapping of the Al-30HEA has shown the formation of a transitional layer having a thickness of ~400 to 500 nm. These transitional layer formed at the interfaces of the Al matrix and non-equiatomic HEA enhances the microhardness to a great extent. The microhardness of Al-30HEA was found to be ~1.72 GPa, and the yield strength was predicted to ~486 MPa. This high values of microhardness and yield strength will help in developing lightweight AMCs for structural applications.

6.2 Suggestions for future work

The present work dealt with the structural transformation, thermal stability and microstructural features of IQC-Sn, Al-IQC and Al-HEA composite processed through mechanical milling and SPS/ HP/ pressure-less sintering. In view of the observation made from the present work and broaden our understanding of these materials, the following suggestions can be made for future investigations:

1. The Al-Cu-Fe IQC reinforced with Sn can be processed through mechanical and cryomilling, and by the only cryomilling. The milling at liquid nitrogen temperature will give an insight into the structural transformation and nanostructuring of the matrix as well as Sn reinforcement.

2. The detailed indentation behaviour and scratch testing of the IQC-Sn SPSed composite and room temperature and high temperature up to 200°C (473 K) can be carried out. This will advocate its suitability as a potential coating material for bearing applications.
3. The cryomilling of Al-IQC and Al-HEA nanocomposite powder and optimization of its processing time may be done. This will help in establishing its effect on the microstructural refinement and mechanical properties.
4. For establishing the effect of transition layer on the mechanical preoperties, the Al-HEA composite can be sintered by SPS. The interface structure and stability requires to be investigated systematically.
5. For studying the effect of interfacial reaction and transition layer on the microhardness, In-situ indentation studies of Al-IQC and Al-HEA SPSed composite may be accomplished in future.
6. Theoretical modelling of mechanical properties of Al-IQC and Al-HEA nanocomposite powder and SPSed sample can be done for ascertaining its correlation with the experimental findings.