Claims

1. A method of forming a non-stoichiometric metal oxide nanoparticle agglomerate, comprising:

a) aerosolizing *via* a nozzle of a burner, a heated mixture comprising a combustible gas mixture having an oxygen-lean equivalence ratio (φ) and a volatile metal oxide precursor, where the combustible gas mixture is ignited at an outlet of the nozzle to provide a flame, such that the volatile metal oxide precursor is decomposed and undergoes rapid nucleation and coagulation to form non-stoichiometric metal oxide nanoparticles in the presence of the flame; and

b) depositing the non-stoichiometric metal oxide nanoparticles on a surface of a substrate positioned at a distance from the nozzle for a period of time for aggregation and oxidation of nanoparticles to form a non-stoichiometric metal oxide nanoparticle agglomerate on the surface of the substrate, wherein:

the oxygen-lean equivalence ratio (φ) of the combustible gas mixture is less than or equal to 1.5; and

a velocity of the combustible gas mixture at the nozzle of the burner is from 300 to 500 cm/s.

2. The method according to Claim 1, wherein the oxygen-lean equivalence ratio (φ) is from 1.05 to 1.5.

3. The method according to Claim 2, wherein the oxygen-lean equivalence ratio (φ) is from 1.1 to 1.4.

4. The method according to any one of the preceding claims, wherein the combustible gas mixture comprises an inert gas (e.g. argon), oxygen and one or both of hydrogen gas and a hydrocarbon gas (e.g. C_2H_4).

5. The method according to Claim 4, wherein the inert gas represents greater than 70 volume% of the combustible gas mixture, with oxygen and one or both of hydrogen gas and the hydrocarbon gas providing the balance.

6. The method according to Claim 5, wherein:
the inert gas provides from 75 to 79.9 volume% of the combustible gas mixture;
oxygen provides from 10 to 17 volume% of the combustible gas mixture; and

hydrogen gas and/or the hydrocarbon gas provide from 5 to 10.1 volume% of the combustible gas mixture.

7. The method according to Claim 6, wherein:
the inert gas provides from 78 to 79.75 volume% of the combustible gas mixture;
oxygen provides from 14.4 to 15 volume% of the combustible gas mixture; and
hydrogen gas and/or the hydrocarbon gas provide from 5.25 to 8 volume% of the combustible gas mixture.

8. The method according to any one of the preceding claims, wherein:
(a) the volatile metal oxide precursor is injected into the heated mixture at a flow rate of from 2 mL/h to 100 mL/h, such as from 3 mL/h to 50 mL/h, such as from 5 mL/h to 25 mL/h, such as from 10 mL/h to 20 mL/h; and/or
(b) the volatile metal oxide precursor has a concentration of from 100 to 1,500 ppm, such as from 250 to 900 ppm, in the combustible gas mixture.

9. The method according to any one of the preceding claims, wherein the flame has a temperature of from 1,500 to 2,500 K.

10. The method according to any one of the preceding claims wherein the substrate is a rotatable substrate, such that when rotated, the deposition of the non-stoichiometric metal oxide nanoparticle agglomerate occurs on only the portion of the substrate within an area under the flame at any given point of time.

11. The method according to Claim 10, wherein the rotatable substrate is rotated at a speed of from 0.1 rpm to 500 rpm, such as from 1 rpm to 300 rpm, or from 1 rpm to 100 rpm, such as 0.1 rpm, 1 rpm, 100 rpm or 300 rpm.

12. The method according to Claim 10 or Claim 11, wherein the deposited non-stoichiometric metal oxide nanoparticle agglomerate is substantially removed from the surface of the rotatable substrate by a collection means or apparatus, such that the deposited non-stoichiometric metal oxide nanoparticle agglomerate is continuously collected from the surface of the rotatable substrate with each rotation, optionally wherein the rotatable substrate is rotated at a speed of from 0.1 rpm to 20 rpm, such as from 0.2 rpm to less than or equal to 5 rpm, such as 1 rpm.

13. The method according to Claim 12, wherein the method is operated for a period of from 1 minute to infinity, such as from 5 minute to 1 month, such as from 15 minutes to 1 week, such as from 25 minutes to 1 hour.

14. The method according to Claim 10, wherein the deposited non-stoichiometric metal oxide nanoparticle agglomerate is deposited on the surface of the rotatable substrate over a period of time of from 1 second to 15 minutes, optionally wherein the rotatable substrate is rotated at a speed of from 100 rpm to 500 rpm, such as from 100 rpm to 300 rpm.

15. The method according to Claim 12 or Claim 13 or the method according to Claim 14, wherein the rotatable substrate is actively cooled after passing through the flame, optionally wherein the rotatable substrate is cooled by a plurality of nozzles of air, each delivering from 20 to 200 L/min, such as from 50 to 150 L/min, such as from 75 to 100 L/min.

16. The method according to any one of the preceding claims, wherein the distance between the nozzle of the burner and the substrate is from 5 to 25 mm, such as from 14 mm to 20 mm, such as greater than 14 mm to 15 mm.

17. The method according to any one of the preceding claims, wherein the substrate has a temperature of from 298 K to 800 K, such as from 350 K to 780K, such as from 400 K to 500 K, optionally wherein the temperature of the substrate is measured at a point outside of the flame.

18. The method according to any one of the preceding claims, wherein the volatile metal oxide precursor comprises a metal that is able to form a stable reducible oxide.

19. The method according to Claim 18, wherein the volatile metal oxide precursor comprises one or more metals selected from the group consisting of Ti, Ce, Co, Fe, Cu and W, optionally wherein the volatile metal oxide precursor comprises Ti.

20. The method according to any one of the preceding claims, wherein one or both of the following apply:

(a) the formed non-stoichiometric metal oxide nanoparticle agglomerate comprises oxygen vacancies, optionally wherein the oxygen vacancies are formed at grain boundaries; and

(b) the deposition in step (b) is carried out in an environment comprising oxygen.