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Reply to Comment on "Preparation of transition metal phosphides using the facile solid state synthesis"

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Peng et al. Reply: The preceding comment raises issues regarding our work [1] which argues whether it is difficult or easy to obtain stoichiometric Co–P compounds in solid state diffusion technique.

They state that (i) stoichiometric transition metal phosphides can be easily synthesized using the solid-state diffusion technique and (ii) the problem of volatilization and sublimation of red phosphorous can be easily tackled. We state that it is difficult to obtain stoichiometric Co–P compounds due to the volatilization and sublimation of red phosphorous [1].

We should emphasize that our statement bases on the comparison of our synthesis method with theirs, which is relative results. We agree that stoichiometric transition metal phosphides could be synthesized using the solid-state diffusion technique and the problem of volatilization and sublimation of red phosphorous could be tackled, so we do not state that it is impossible or other similar descriptions. But compared with ours, their synthesis process is relatively difficult and complicated to proceed, with detailed information as follows:

Firstly, there was pretreatment process in the synthesis of $(Co_{1-x}Fe_x)_2P$ compounds in the related papers [2–8]. As described in the comment, the surfaces of transition metal powders were reduced by heating them in hydrogen atmosphere at ~700 °C for 24 h. And a specially designed tubular furnace was also needed, with N_2 gas flushing for an hour and then continuous flow of H_2 gas. However, there was no need in our paper.

Secondly, there was strict adding order for reactants in the synthesis of $(Co_{1-x}Fe_x)_2P$ compounds [2–8]. It was essential to keep the red phosphorous at the bottom of the ampoule, which were fully covered by metal powders. Otherwise, during the heating, the phosphorous vapour would move up and may get deposited on the inner wall of the upper half of the quartz ampoule, which did not produce stoichiometric products due to phosphorous deficiency. But the mixing process of reactants was simple and facile in our paper [1]—the reactants were all dissolved in distilled water for a transparent solution, which was then placed in an oven until the water was evaporated.

Thirdly, as described in the comment, the ampoules should be vacuum (better than 10^{-3} Torr) sealed to avoid the oxidation of the reactants. However, there was no need in our paper.

Fourthly, as described in the comment, the temperature of the furnace was kept at 250 °C for the first 72 h, raised in steps over a period of 7 days to 1000 °C, and maintained for 15 days. Rapid heating around melting temperature (~280 °C) of red phosphorous may induce an abrupt explosion inside the ampoule, which resulted in the breaking of the ampoule. After this, the ampoule was rapidly quenched in distilled water at room temperature. The obtained products were resealed in evacuated ampoules at 1000 °C for another 15 days. But in our synthesis route, the obtained pink powders were merely calcined at 600 °C for 6 h at a heating rate of 10 K min^{-1} in inert Ar atmosphere [1].

The preceding comment may be a misunderstanding of our statements. We cite the papers to speak highly of their contributions to solid-state diffusion technique in the synthesis of transition metal phosphides. We state that it is difficult rather than impossible or other similar words, which are relative results compared the related synthesis routes with ours. Because the reported synthesis process is complicated and strict but ours is simple and facile to be carried out.

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